STRUCTURAL CHEMISTRY
ISSN 2053-2296

4-Styrylquinolines from cyclocondensation reactions between (2-aminophenyl)chalcones and 1,3-diketones: crystal structures and regiochemistry

Diego Rodríguez,[a] Sergio Andrés Guerrero,[a] Alirio Palma,[a] Justo Cobo[b] and Christopher Glidewell[c]*

[a]Laboratorio de Síntesis Orgánica, Escuela de Química, Universidad Industrial de Santander, AA 678, Bucaramanga, Colombia, [b]Departamento de Química Inorgánica y Orgánica, Universidad de Jaén, 23071 Jaén, Spain, and [c]School of Chemistry, University of St Andrews, Fife KY16 9ST, Scotland. *Correspondence e-mail: cg@st-andrews.ac.uk

Received 3 August 2020
Accepted 4 August 2020

Edited by A. L. Spek, Utrecht University, The Netherlands

Keywords: synthesis; cyclocondensation; quinolone; crystal structure; molecular conformation; hydrogen bonding; supramolecular assembly; regiochemistry.

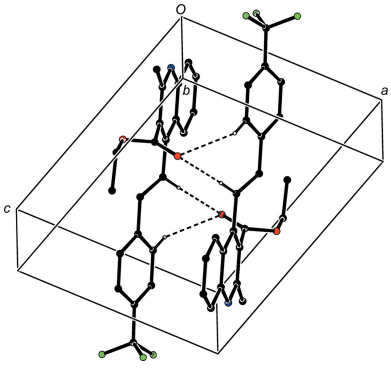
CCDC references: 2021528; 2021527; 2021526; 2021525; 2021524; 2021523

Supporting information: this article has supporting information at journals.iucr.org/c

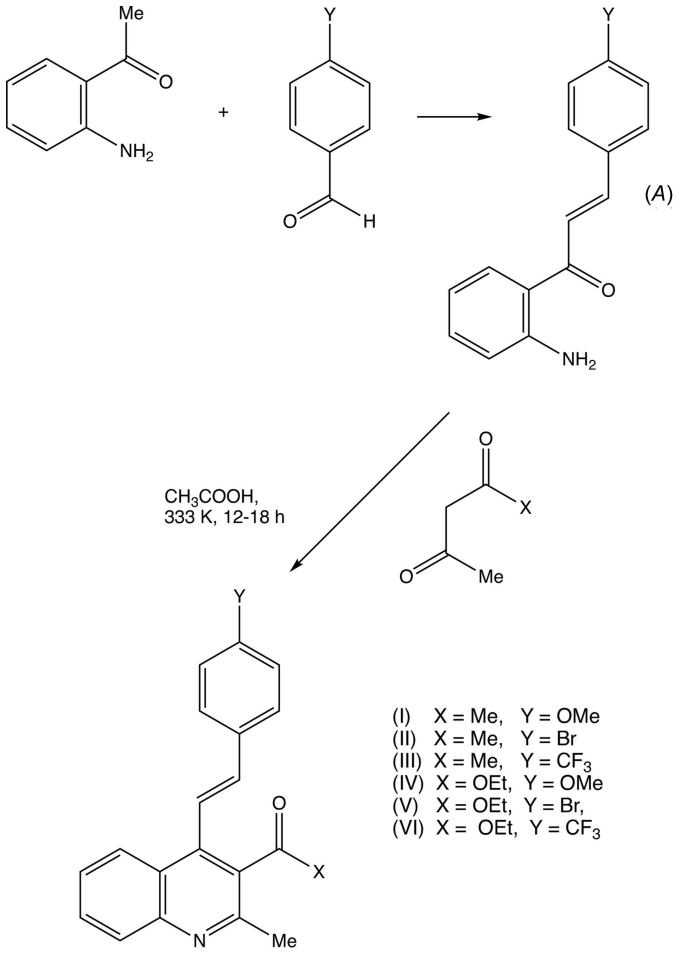
Structures are reported for two matched sets of substituted 4-styrylquinolines which were prepared by the formation of the heterocyclic ring in cyclocondensation reactions between 1-(2-aminophenyl)-3-arylprop-2-en-1-ones with 1,3-dicarbonyl compounds. (*E*)-3-Acetyl-4-[2-(4-methoxyphenyl)ethenyl]-2-methylquinoline, $C_{21}H_{19}NO_2$, (I), (*E*)-3-acetyl-4-[2-(4-bromophenyl)ethenyl]-2-methylquinoline, $C_{20}H_{16}BrNO$, (II), and (*E*)-3-acetyl-2-methyl-4-{2-[4-(trifluoromethyl)phenyl]ethenyl}quinoline, $C_{21}H_{16}F_3NO$, (III), are isomorphous and in each structure the molecules are linked by a single C—H···O hydrogen bond to form *C*(6) chains. In (I), but not in (II) or (III), this is augmented by a C—H···π(arene) hydrogen bond to form a chain of rings; hence, (I)–(III) are not strictly isostructural. By contrast with (I)–(III), no two of ethyl (*E*)-4-[2-(4-methoxyphenyl)ethenyl]-2-methylquinoline-3-carboxylate, $C_{22}H_{21}NO_3$, (IV), ethyl (*E*)-4-[2-(4-bromophenyl)ethenyl]-2-methylquinoline-3-carboxylate, $C_{21}H_{18}BrNO_2$, (V), and ethyl (*E*)-2-methyl-4-{2-[4-(trifluoromethyl)phenyl]ethenyl}-quinoline-3-carboxylate, $C_{22}H_{18}F_3NO_2$, (VI), are isomorphous. The molecules of (IV) are linked by a single C—H···O hydrogen bond to form *C*(13) chains, but cyclic centrosymmetric dimers are formed in both (V) and (VI). The dimer in (V) contains a C—H···π(pyridyl) hydrogen bond, while that in (VI) contains two independent C—H···O hydrogen bonds. Comparisons are made with some related structures, and both the regiochemistry and the mechanism of the heterocyclic ring formation are discussed.

1. Introduction

Compounds containing 2-styrylquinoline units have attracted interest in recent years because of their potential as anticancer (El-Sayed *et al.*, 2018), anti-HIV (Polanski *et al.*, 2002), antimalarial (Roberts *et al.*, 2017) and antimicrobial (Cieslik *et al.*, 2012) agents, as well as in the treatment of Alzheimer's dementia (Wang *et al.*, 2015). By contrast, analogous compounds containing 4-styryl units have been very much less extensively investigated, probably, at least in part, because of a lack of efficient and versatile methods for their synthesis: such methods have generally been based on coupling reactions requiring the prior synthesis of haloquinolines or (haloalkyl)-quinolines, combined with either harsh reaction conditions or the use of expensive heavy-metal catalysts (Omar & Hormi, 2009; Xia *et al.*, 2016). However, a very straightforward synthesis of 4-styrylquinolines has been developed recently (Meléndez *et al.*, 2020), in which the heterocyclic ring of the quinoline unit is built *in situ* using a cyclocondensation reaction between a 2′-aminochalcone, (*A*), and a 1,3-dicarbonyl compound (*cf.* Scheme 1). The chalcone component in this

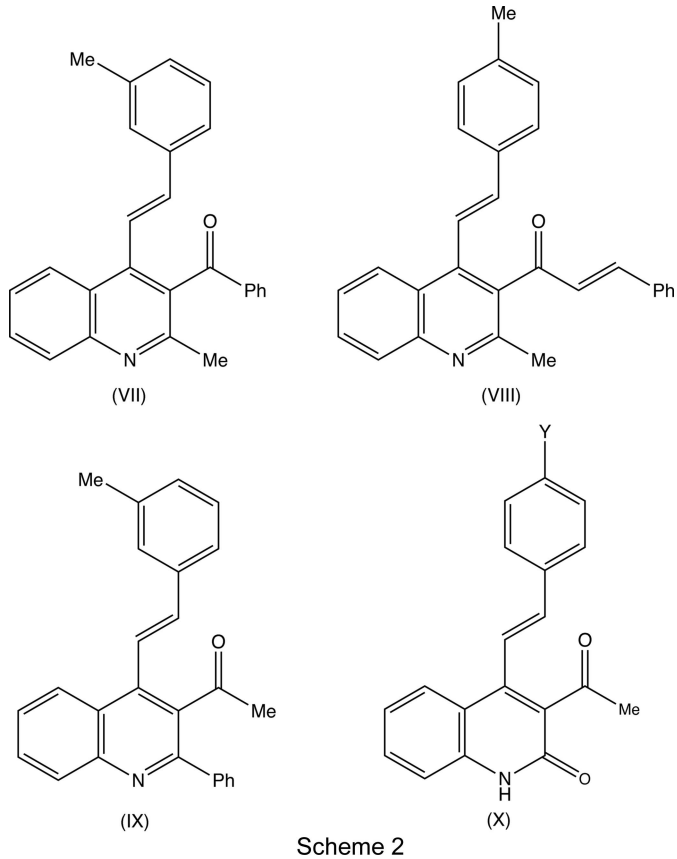


type of cyclization is readily accessible by reaction between 2′-aminoacetophenone and an aromatic aldehyde, allowing incorporation of a wide variety of substituents both in the styryl portion and at the 3-position of the quinoline nucleus. We report here the molecular structures and supramolecular assembly of two matched sets, each of three related products: the 3-acetyl derivatives, compounds (I)–(III) (Scheme 1 and Figs. 1–3), where X = Me, were all obtained using pentane-2,4-dione as the dicarbonyl component, while the 3-carboethoxy derivatives, compounds (IV)–(VI) (Figs. 4–6), where X = OEt, were all obtained using ethyl 3-oxobutanoate (ethyl acetoacetate). Compounds such as (I)–(III), containing an acetyl



Scheme 1

group, can act as useful synthetic intermediates, as they can undergo condensation with a further substituted aldehyde to form a chalcone substituent at the 3-position, as exemplified by compound (VIII) (Scheme 2). Such chalcones can themselves then undergo cyclocondensation reactions, for example, with a hydrazine, to form either a pyrazole, under basic conditions (Samshuddin *et al.*, 2014), or a reduced pyrazole ring, under acidic conditions (Jasinski *et al.*, 2010), or with guanidine to form a reduced pyrimidine ring (Nayak *et al.*, 2014), thus giving access to a rich diversity of new 4-styrylquinolin-3-yl heterocycles. In addition to reporting the molecular and supramolecular structures of compounds (I)–(VI), we also briefly consider compounds (VII) and (VIII) (Scheme 2). These have been reported on a simple proof of constitution basis [Cambridge Structural Database (CSD; Groom *et al.*, 2016) refcodes MUMZEC and MUMZIG (Meléndez *et al.*, 2020)] but without any structure description or discussion; accordingly, we discuss here the supramolecular assembly in these two compounds.



Scheme 2

2. Experimental

2.1. Synthesis and crystallization

Samples of compounds (I)–(VI) were prepared and crystallized following a recently published procedure (Meléndez *et al.*, 2020).

2.2. Refinement

Crystal data, data collection and structure refinement details for compounds (I)–(VI) are summarized in Table 1. Two low-angle reflections which had been attenuated by the beam stop [100 for (I) and $\bar{1}$01 for (VI)] were omitted from the data sets before the final refinements; likewise, two bad outlier reflections (639 and 606) were removed from the data set for (IV). All H atoms were located in difference maps and subsequently treated as riding atoms in geometrically idealized positions, with C—H = 0.95 (alkenyl, aromatic and heteroaromatic), 0.98 (CH_3) or 0.99 Å (CH_2), and with

Table 1
Experimental details.

Experiments were carried out at 100 K with Mo $K\alpha$ radiation using a Bruker D8 Venture diffractometer. Absorption was corrected for by multi-scan methods (*SADABS*; Bruker, 2016). H-atom parameters were constrained.

	(I)	(II)	(III)
Crystal data			
Chemical formula	$C_{21}H_{19}NO_2$	$C_{20}H_{16}BrNO$	$C_{21}H_{16}F_3NO$
M_r	317.37	366.24	355.35
Crystal system, space group	Monoclinic, $P2_1/c$	Monoclinic, $P2_1/c$	Monoclinic, $P2_1/c$
a, b, c (Å)	8.2595 (4), 6.4279 (3), 31.9064 (14)	8.0849 (3), 6.6692 (2), 31.1063 (10)	8.0822 (4), 6.6567 (4), 32.1024 (17)
α, β, γ (°)	90, 93.674 (2), 90	90, 95.005 (1), 90	90, 90.576 (2), 90
V (Å^3)	1690.47 (14)	1670.85 (10)	1727.05 (16)
Z	4	4	4
μ (mm^{-1})	0.08	2.46	0.11
Crystal size (mm)	0.23 × 0.16 × 0.09	0.17 × 0.11 × 0.04	0.20 × 0.08 × 0.06
Data collection			
T_{min}, T_{max}	0.947, 0.993	0.810, 0.906	0.942, 0.994
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	39800, 4197, 3528	38342, 3839, 3434	19648, 3959, 3363
R_{int}	0.051	0.034	0.032
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.668	0.650	0.650
Refinement			
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.040, 0.111, 1.06	0.030, 0.073, 1.03	0.041, 0.101, 1.04
No. of reflections	4197	3839	3959
No. of parameters	220	210	237
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.33, −0.23	0.56, −0.76	0.28, −0.23

	(IV)	(V)	(VI)
Crystal data			
Chemical formula	$C_{22}H_{21}NO_3$	$C_{21}H_{18}BrNO_2$	$C_{22}H_{18}F_3NO_2$
M_r	347.40	396.26	385.37
Crystal system, space group	Triclinic, $P\overline{1}$	Monoclinic, $P2_1/n$	Triclinic, $P\overline{1}$
a, b, c (Å)	9.5301 (8), 10.3513 (8), 10.3621 (8)	9.5709 (6), 10.6119 (7), 18.2074 (10)	8.7465 (10), 9.9436 (11), 11.1116 (11)
α, β, γ (°)	65.374 (3), 86.583 (3), 76.376 (3)	90, 90.939 (2), 90	105.446 (4), 99.763 (4), 97.204 (4)
V (Å^3)	902.23 (13)	1849.0 (2)	903.08 (17)
Z	2	4	2
μ (mm^{-1})	0.09	2.24	0.11
Crystal size (mm)	0.30 × 0.12 × 0.05	0.25 × 0.18 × 0.15	0.27 × 0.20 × 0.18
Data collection			
T_{min}, T_{max}	0.954, 0.996	0.595, 0.715	0.954, 0.980
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	44489, 4158, 3440	54534, 4588, 4098	59876, 4491, 3692
R_{int}	0.053	0.041	0.042
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.650	0.667	0.667
Refinement			
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.052, 0.128, 1.07	0.022, 0.056, 1.02	0.044, 0.120, 1.05
No. of reflections	4158	4588	4491
No. of parameters	238	228	255
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.36, −0.21	0.35, −0.42	0.43, −0.43

Computer programs: *APEX3* (Bruker, 2018), *SAINT* (Bruker, 2017), *SHELXT2014* (Sheldrick, 2015*a*), *SHELXL2014* (Sheldrick, 2015*b*) and *PLATON* (Spek, 2020).

$U_{iso}(H) = kU_{eq}(C)$, where $k = 1.5$ for the methyl groups, which were permitted to rotate but not to tilt, and 1.2 for all other H atoms. For compounds (VII) and (VIII), the published structures (Meléndez *et al.*, 2020) were inverted and the atom labelling adjusted slightly in order to bring them into full conformity with compounds (I)–(VI) (*cf.* Tables 2 and 3); the modified versions of the CIFs for (VII) and (VIII) are provided in the supporting information. Examination of the structure for (VIII) using *PLATON* (Spek, 2020) showed that the unit cell contains two voids, each of volume 60 Å^3 and centred at (0, $\frac{1}{2}$, 0) and ($\frac{1}{2}$, 0, $\frac{1}{2}$), but further examination using SQUEEZE (Spek, 2015) showed that these voids contained negligible electron density.

3. Results and discussion

In reactions between a chalcone of type (*A*) (Scheme 1) and a symmetrical 1,3-diketone, such as pentane-2,4-dione, only a single product is possible, namely, the 3-acetyl-2-methylquinoline derivative, as exemplified by compounds (I)–(III). However, a comparable reaction involving an unsymmetrical diketone, such as 1-phenylbutane-1,3-dione can give two

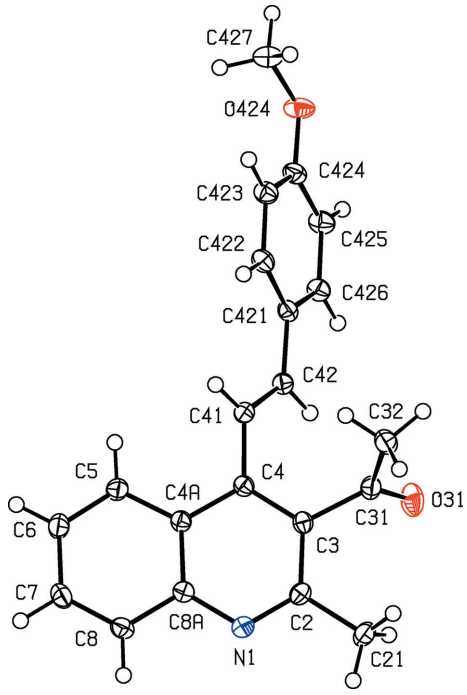


Figure 1
The molecular structure of compound (I), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 50% probability level.

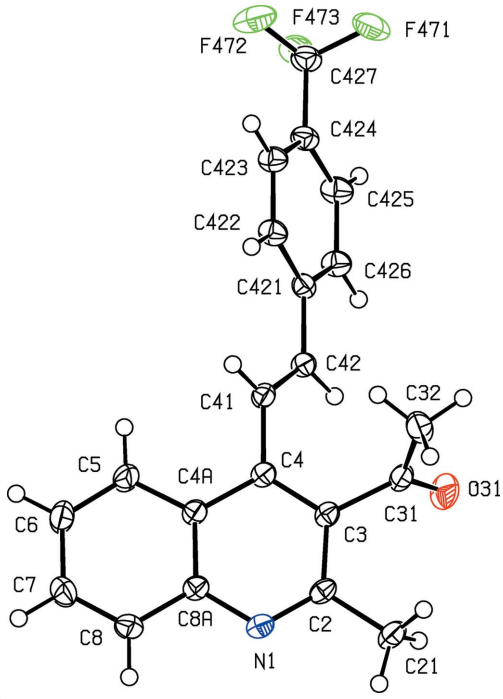


Figure 3
The molecular structure of compound (III), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 50% probability level.

regioisomers, such as (VII), if the amino group reacts at the acetyl carbonyl group, or the alternative (IX) if the reaction occurs at the benzoyl carbonyl group. In general, reactions with this ketone lead exclusively to the 3-benzoyl-2-methyl isomers, as exemplified by (VII), rather than to the 3-acetyl-2-phenyl alternative exemplified by (IX) (Meléndez *et al.*, 2020), which is consistent with the greater reactivity in the nucleophilic addition reaction of acetyl groups compared with benzoyl groups (Bürgi *et al.*, 1974; Katritzsky *et al.*, 1995; Meléndez *et al.*, 2020). Similarly, the reaction of a chalcone of type (*A*) with an unsymmetrical diketo compound, such as

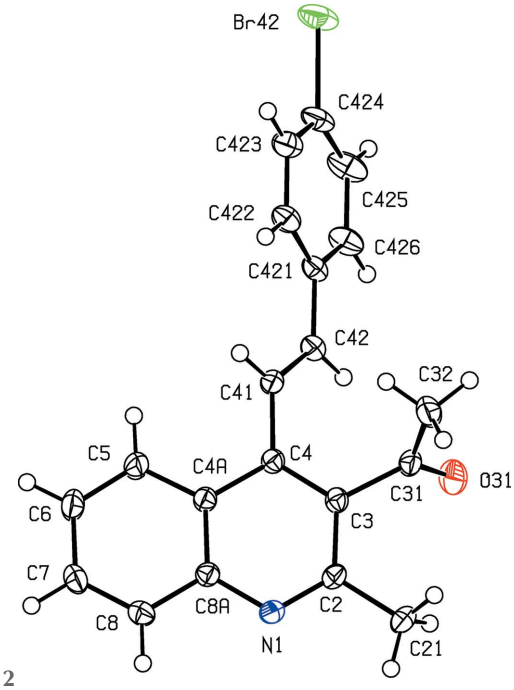


Figure 2
The molecular structure of compound (II), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 50% probability level.

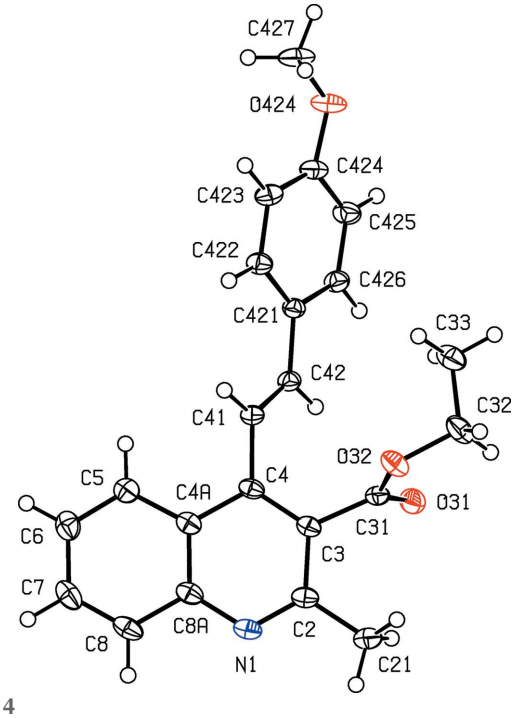


Figure 4
The molecular structure of compound (IV), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 50% probability level.

Table 2
Selected torsion angles (°) for compounds (I)–(VIII).

Compound	C3—C4—C41—C42	C41—C42—C421—C422	C2—C3—C31—O31	C2—C3—C31—O32
(I)	46.40 (16)	13.78 (18)	66.18 (15)	
(II)	46.8 (3)	14.8 (3)	68.2 (2)	
(III)	49.0 (2)	13.0 (2)	69.52 (18)	
(IV)	48.8 (2)	1.2 (3)	73.7 (2)	−104.85 (17)
(V)	51.4 (2)	10.6 (2)	−102.56 (17)	75.88 (15)
(VI)	34.8 (2)	−17.7 (2)	−99.75 (16)	76.46 (15)
(VII)	51.0 (5)	2.0 (5)	71.5 (4)	
(VIII)	54.17 (19)	−4.0 (2)	−97.59 (15)	

ethyl 3-oxobutanoate, can, in principle, give two types of product: reaction of the amino group at the acetyl carbonyl group leads to ethyl esters, as exemplified by compounds (IV)–(VI), but reaction of the amino group at the ester carbonyl group would lead to elimination of ethanol with the formation of a 2-quinolone of type (X) (Scheme 2). Again, these reactions appear to lead exclusively to the esters, as exemplified by (IV)–(VI) (Meléndez *et al.*, 2020), consistent with the greater electrophilicity of a ketonic carbonyl group compared with an ester carbonyl group. On the other hand, 2-aryl-4-quinolones are sometimes formed as by-products arising from an intramolecular cyclization of the chalcone precursor.

Compounds (I)–(III), where X = Me and Y = OMe, Br or CF_3, respectively (Scheme 1 and Figs. 1–3), all crystallize in the space group $P2_1/c$ with rather similar unit-cell dimensions (Table 1) and very similar molecular conformations (Table 2); each structure can be refined using the coordinates of one of the others as the starting point, provided due alteration is made in the substituent at atom C424 (Figs. 1–3). However, although there are short intermolecular C—H···O and C—H···π(arene) contacts in all three compounds, involving the same sets of atoms (Table 3), in each of compounds (II) and

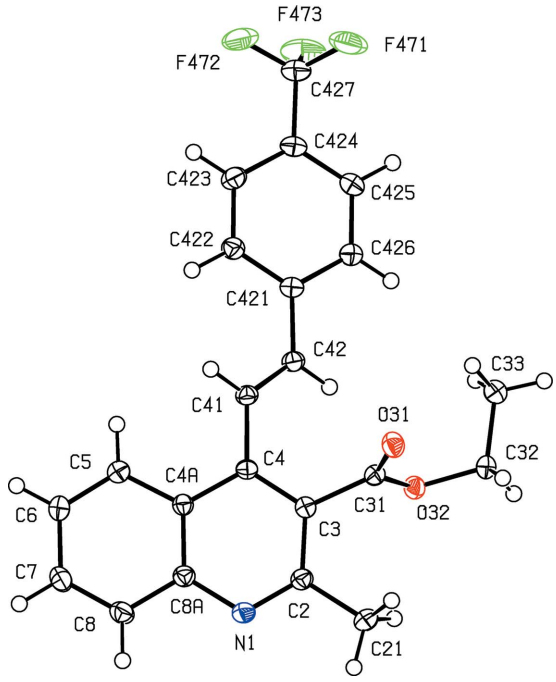


Figure 6
The molecular structure of compound (VI), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 50% probability level.

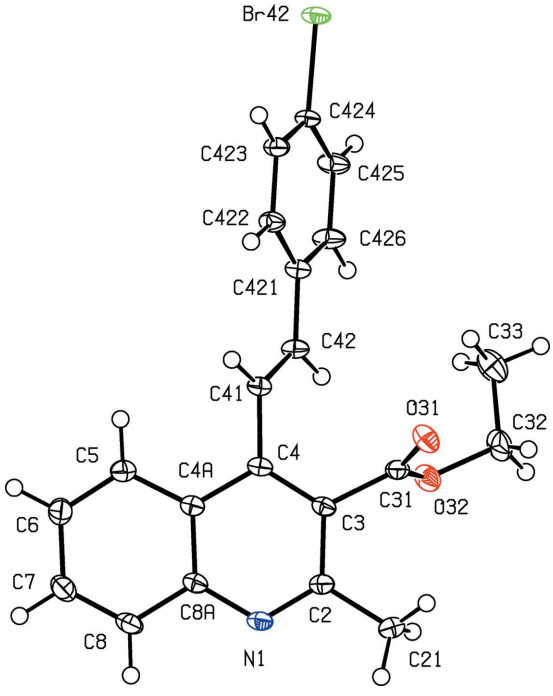


Figure 5
The molecular structure of compound (V), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 50% probability level.

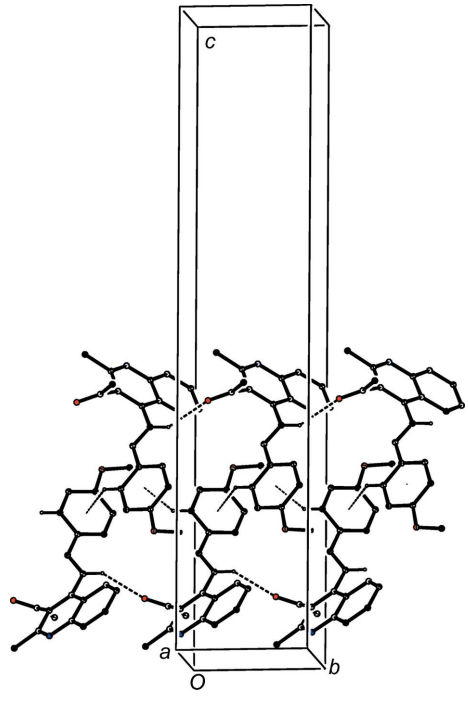


Figure 7
Part of the crystal structure of compound (I), showing the formation of a chain of rings along [010] built from C—H···O and C—H···π(arene) hydrogen bonds, drawn as dashed lines. For the sake of clarity, H atoms not involved in the motifs shown have been omitted.

Table 3
Hydrogen bonds and short intramolecular contacts (Å, °) for compounds (I)–(VIII).

*Cg*1, *Cg*3, *Cg*4 and *Cg*5 represent the centroids of the N1/C2–C4/C4*A*/C8*A*, C421–C426, C311–C316 [present in (VII) only] and C331–C336 [present in (VIII) only] rings, respectively; ring 2 comprises atoms C4*A*/C5–C8/C8*A*.

	D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
(I)	C41—H41···O31^{i}	0.95	2.41	3.2527 (15)	148
	C426—H426···*Cg*3^{ii}	0.95	2.77	3.5252 (13)	138
(II)	C41—H41···O31^{i}	0.95	2.37	3.283 (2)	161
	C426—H426···*Cg*3^{ii}	0.95	2.91	3.7071 (19)	142
(III)	C41—H41···O31^{i}	0.95	2.42	3.3290 (17)	161
	C426—H426···*Cg*3^{ii}	0.95	3.00	3.7621 (15)	138
(IV)	C32—H32*B*···O424^{iii}	0.99	2.57	3.406 (2)	142
	C423—H423···*Cg*1^{iv}	0.95	2.91	3.4894 (19)	120
(V)	C423—H423···O31^{v}	0.95	2.59	3.2197 (17)	124
	C426—H426···*Cg*1^{iv}	0.95	2.74	3.5961 (16)	151
(VI)	C41—H41···O31^{iv}	0.95	2.54	3.4922 (18)	178
	C422—H422···O31^{iv}	0.95	2.56	3.3924 (19)	146
(VII)	C41—H41···O31^{vi}	0.95	2.37	3.300 (5)	167
	C422—H422···O31^{vi}	0.95	2.57	3.506 (4)	169
	C426—H426···*Cg*4^{vii}	0.95	2.67	3.549 (4)	155
(VIII)	C334—H334···O31^{viii}	0.95	2.61	3.542 (2)	168
	C7—H7···*Cg*5^{ix}	0.95	2.93	3.6437 (16)	133
	C422—H422···*Cg*1^{x}	0.95	2.93	3.6300 (17)	132

Symmetry codes: (i) $x, y+1, z$; (ii) $-x+1, y-\frac{1}{2}, -z+\frac{1}{2}$; (iii) $x+1, y, z$; (iv) $-x+1, -y+1, -z+1$; (v) $-x+\frac{3}{2}, y+\frac{1}{2}, -z+\frac{3}{2}$; (vi) $x-\frac{1}{2}, y, -z+\frac{1}{2}$; (vii) $-x+\frac{3}{2}, y-\frac{1}{2}, z$; (viii) $x-1, y, z$; (ix) $x+\frac{1}{2}, -y+\frac{1}{2}, z-\frac{1}{2}$; (x) $x+\frac{1}{2}, -y+\frac{1}{2}, z+\frac{1}{2}$.

(III), the H···*Cg* distance is quite long and probably of marginal structural significance, whereas it can be regarded as a genuine hydrogen bond in compound (I). On this basis, compounds (I)–(III) can be regarded as isomorphous but not strictly isostructural (Acosta *et al.*, 2009; Blanco *et al.*, 2012). However, in the comparable series of compounds, *i.e.* (IV)–(VI), where *X* = OEt, although compounds (IV) and (VI) are both triclinic, in (IV) the inter-axial angles are all less than 90°, but in (VI) they are greater than 90°, so that these two compounds are far from being isomorphous. On the other hand, the third member of this group, compound (V), is monoclinic, so there can be no close similarities within this group.

None of the molecules of (I)–(VIII) exhibits any internal symmetry, so that they are all conformationally chiral; in each case, the reference molecule was selected as one having a positive sign for the C3—C4—C41—C42 torsion angle (Table 2), although the space groups confirm that all the compounds have crystallized as conformational racemates.

The supramolecular assembly of compounds (I)–(VI) is determined by C—H···O and C—H···π hydrogen bonds (Table 3). In each of (I)–(III), molecules which are related by translation are linked by a C—H···O hydrogen bond to form a *C*(6) (Etter, 1990; Etter *et al.*, 1990; Bernstein *et al.*, 1995) chain running parallel to the [010] direction. In the structure of (I), this is enhanced by a C—H···π(arene) hydrogen bond linking molecules related by the 2_1 screw axis along $(\frac{1}{2}, y, \frac{1}{4})$ to form a chain of rings (Fig. 7). However, in the structures of (II) and (III), the corresponding H···*Cg* and C···*Cg* distances are much longer than they are in (I), so that these are possibly better regarded as short adventitious contacts rather than structurally significant hydrogen bonds.

A single C—H···O hydrogen bond links the molecules of compound (IV) which are related by translation into a *C*(13) chain running parallel to the [100] direction (Fig. 8). This

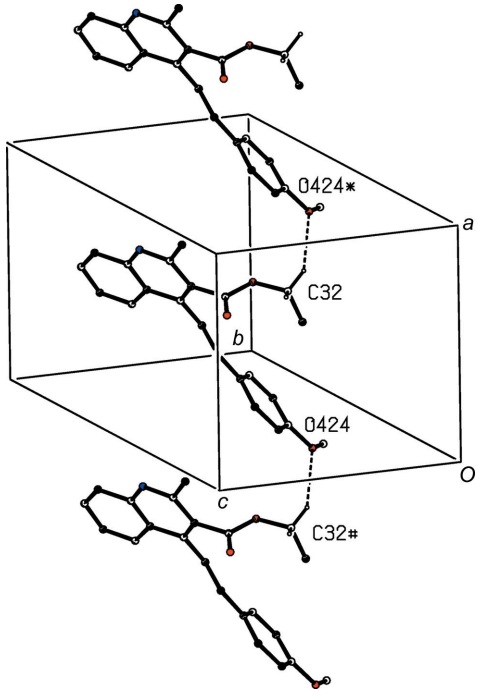


Figure 8
Part of the crystal structure of compound (IV), showing the formation of a *C*(13) chain running parallel to the [100] direction. Hydrogen bonds are drawn as dashed lines and, for the sake of clarity, H atoms bonded to those C atoms which are not involved in the motif shown have been omitted. Atoms marked with an asterisk (*) or a hash (#) are at the symmetry positions $(x+1, y, z)$ and $(x-1, y, z)$, respectively.

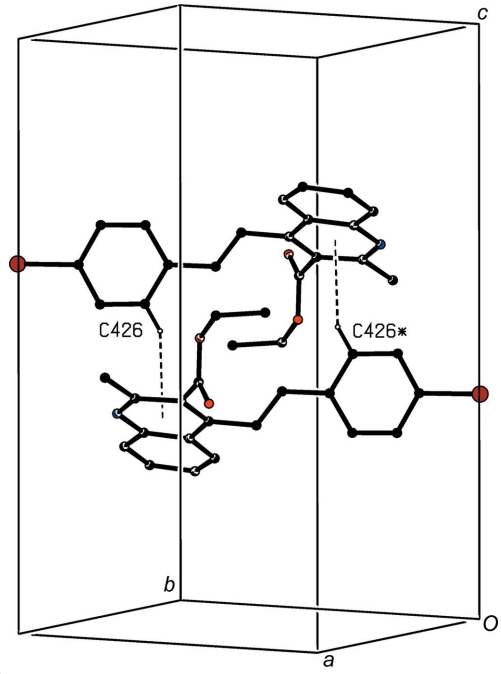


Figure 9
Part of the crystal structure of compound (V), showing the formation of a centrosymmetric dimer. Hydrogen bonds are drawn as dashed lines and, for the sake of clarity, H atoms which are not involved in the motif shown have been omitted. The atom marked with an asterisk (*) is at the symmetry position $(-x+1, -y+1, -z+1)$.

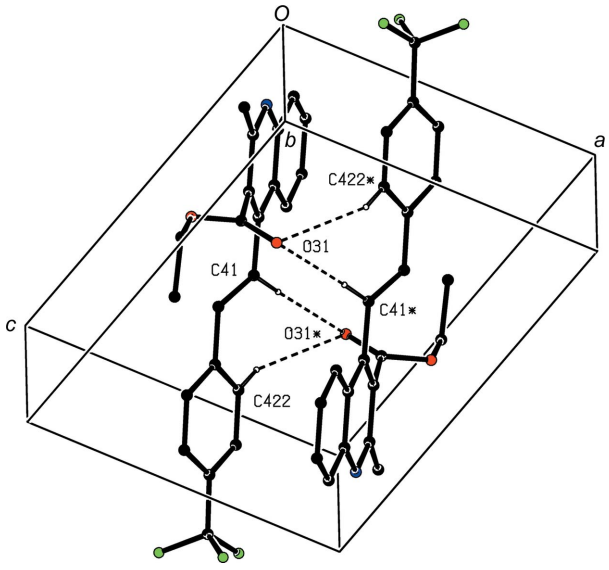


Figure 10
Part of the crystal structure of compound (VI), showing the formation of a centrosymmetric dimer. Hydrogen bonds are drawn as dashed lines and, for the sake of clarity, H atoms which are not involved in the motif shown have been omitted. Atoms marked with an asterisk (*) are at the symmetry position $(-x+1, -y+1, -z+1)$.

structure also contains a short C—H···π(pyridyl) contact, but the long H···*Cg* distance and the very small C—H···*Cg* angle indicate that this is probably not structurally significant (Wood *et al.*, 2009). By contrast, in the structure of compound (V), it is the C—H···O contact which has a very small *D*—H···*A* angle (Table 3), while a C—H···π(pyridyl) hydrogen bond links

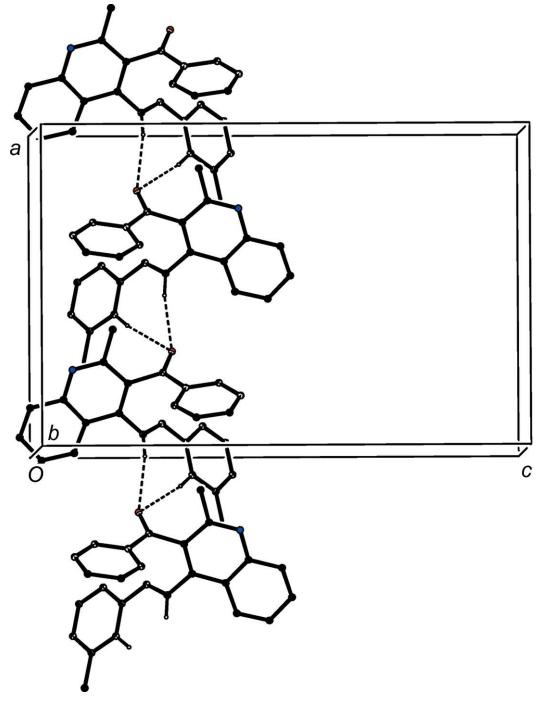


Figure 11
Part of the crystal structure of compound (VII), showing the formation of a $C(6)C(9)[R_2^1(7)]$ chain of rings running parallel to the [100] direction. Hydrogen bonds are drawn as dashed lines and, for the sake of clarity, H atoms which are not involved in the motif shown have been omitted.

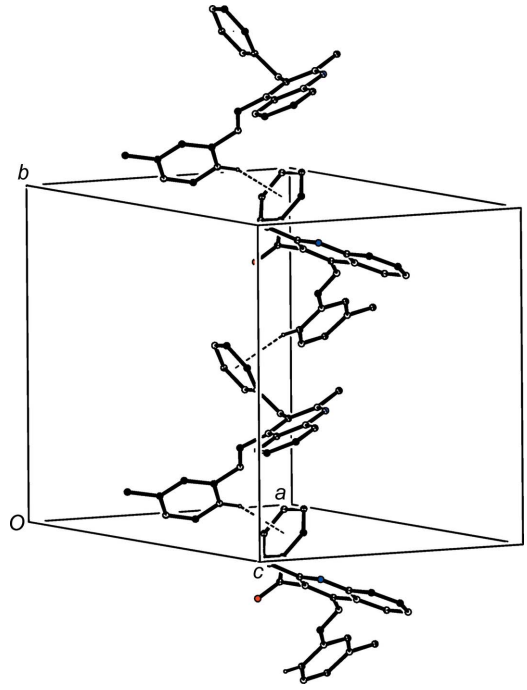


Figure 12
Part of the crystal structure of compound (VII), showing the formation of a chain built from C—H···π(arene) hydrogen bonds, drawn as dashed lines, running parallel to the [010] direction. For the sake of clarity, H atoms which are not involved in the motif shown have been omitted.

molecules which are related by inversion to form a cyclic centrosymmetric dimer (Fig. 9).

In the structure of compound (VI), there are no C—H···π hydrogen bonds or short intermolecular contacts. Instead two C—H···O hydrogen bonds combine to link inversion-related pairs of molecules into centrosymmetric dimers. The hydrogen bonds involving atoms of type C41 form an $R_2^2(12)$ ring, while those involving atoms of type C422 generate an $R_2^2(18)$ ring (Fig. 10).

We also discuss here the supramolecular assembly of compounds (VII) and (VIII), which, as noted above (§1, *Introduction*), have been reported on a simple proof of constitution basis, without discussion (Meléndez *et al.*, 2020). The assembly in (VII) in the space group *Pbcn* is based upon two C—H···O hydrogen bonds and one C—H···π(arene) hydrogen bond (Table 3). The two C—H···O hydrogen bonds link molecules which are related by the *a*-glide plane at $z = \frac{1}{4}$ to form a $C(6)C(9)[R_2^1(7)]$ chain of rings running parallel to the [100] direction (Fig. 11). In addition, the structure of (VII) contains a C—H···π(arene) hydrogen bond which links molecules which are related by the *b*-glide plane at $x = \frac{3}{4}$ to form a chain running parallel to the [010] direction (Fig. 12). The combination of the chain motifs along [100] and [010] generates a complex sheet lying parallel to (001) in the domain $0 < z < \frac{1}{2}$; a second sheet of this type, related to the first by inversion, lies in the domain $\frac{1}{2} < z < 1.0$, but there are no direction-specific interactions between adjacent sheets. Even the shortest intermolecular contacts (Table 3) in chalcone (VIII) have H···*A* distances which are probably too long for these contacts to be regarded as structurally significant.

The structures of several simple 2-styrylquinolines have been published, including those of the unsubstituted 2-styrylquinoline itself (Valle *et al.*, 1986), and of several analogues carrying simple substituents in the phenyl ring (Kuz'mina *et al.*, 2012). In addition, structures have been reported for a number of salts derived from 2-styrylquinolines (Kobkeatthawin *et al.*, 2008, 2009; Chantrapromma *et al.*, 2008, 2014; Fun *et al.*, 2013). For all of these compounds, the styryl group was introduced into a preformed quinoline nucleus. 8-Styrylquinoline and its 4-phenylstyryl analogue, whose structures have also been reported (Sharma *et al.*, 2015), were prepared using a rhodium-catalysed coupling reaction between quinoline *N*-oxide and the styrene component. Despite the substantial number of structure reports involving 2-styrylquinolines and their derivatives, there are no reports in the CSD of 4-styrylquinolines other than the two examples discussed above, *i.e.* compounds (VII) and (VIII) (CSD refcodes MUMZEC and MUMZIG, respectively; Meléndez *et al.*, 2020).

Acknowledgements

The authors thank the Centro de Instrumentación, Científico-Técnica of the Universidad de Jaén (UJA), and its staff for the data collection.

Funding information

Funding for this research was provided by: Vicerrectoría de Investigación y Extensión de la Universidad Industrial de Santander (proyecto No. 2497, to AP); Universidad de Jaén, the Consejería de Economía, Innovación, Ciencia y Empleo (Junta de Andalucía, Spain) and Centro de Instrumentación Científico–Técnica of the Universidad de Jaén (UJA) (to JC).

References

Acosta, L. M., Bahsas, A., Palma, A., Cobo, J., Hursthouse, M. B. & Glidewell, C. (2009). *Acta Cryst.* C**65**, o92–o96.
Bernstein, J., Davis, R. E., Shimoni, L. & Chang, N.-L. (1995). *Angew. Chem. Int. Ed. Engl.* **34**, 1555–1573.
Blanco, M. C., Palma, A., Cobo, J. & Glidewell, C. (2012). *Acta Cryst.* C**68**, o195–o198.
Bruker (2016). *SADABS*. Bruker AXS Inc., Madison, Wisconsin, USA.
Bruker (2017). *SAINT*. Bruker AXS Inc., Madison, Wisconsin, USA.
Bruker (2018). *APEX3*. Bruker AXS Inc., Madison, Wisconsin, USA.
Bürgi, H. B., Dunitz, J. D., Lehn, J. M. & Wipff, M. (1974). *Tetrahedron*, **30**, 1563–1572.
Chantrapromma, S., Kaewmanee, N., Boonnak, N., Quah, C. K. & Fun, H.-K. (2014). *Acta Cryst.* E**70**, o395–o396.
Chantrapromma, S., Kobkeatthawin, T., Chanawanno, K., Karalai, S. & Fun, H.-K. (2008). *Acta Cryst.* E**64**, o876–o877.
Cieslik, W., Musiol, R., Nycz, J. E., Jampilek, J., Vejsova, M., Wolff, M., Machura, B. & Polanski, J. (2012). *Bioorg. Med. Chem.* **20**, 6960–6968.
El-Sayed, M. A.-A., El-Husseiny, W. M., Abdel-Aziz, N. I., El-Azab, A. S., Abuelizz, H. A. & Abdel-Aziz, A. A.-M. (2018). *J. Enzyme Inhib. Med. Chem.* **33**, 199–209.
Etter, M. C. (1990). *Acc. Chem. Res.* **23**, 120–126.
Etter, M. C., MacDonald, J. C. & Bernstein, J. (1990). *Acta Cryst.* B**46**, 256–262.
Fun, H.-K., Kaewmanee, N., Chanawanno, K., Boonnak, N. & Chantrapromma, S. (2013). *Acta Cryst.* E**69**, o1510–o1511.
Groom, C. R., Bruno, I. J., Lightfoot, M. P. & Ward, S. C. (2016). *Acta Cryst.* B**72**, 171–179.
Jasinski, J. P., Guild, C. J., Samshuddin, S., Narayana, B. & Yathirajan, H. S. (2010). *Acta Cryst.* E**66**, o1948–o1949.
Katritzsky, A. R., Meth-Cohn, O. & Rees, C. (1995). In *Comprehensive Organic Functional Group Transformations*. Oxford: Pergamon Press.
Kobkeatthawin, T., Ruanwas, P., Chantrapromma, S. & Fun, H.-K. (2008). *Acta Cryst.* E**64**, o642–o643.
Kobkeatthawin, T., Suwunwong, T., Chantrapromma, S. & Fun, H.-K. (2009). *Acta Cryst.* E**65**, o76–o77.
Kuz'mina, L. G., Sitin, A. G., Gulakova, E., Fedorova, O. A., Lermontova, E. K. & Churakov, A. V. (2012). *Crystallogr. Rep.* **57**, 85–95.
Meléndez, A., Plata, A., Rodríguez, R., Ardila, D., Guerrero, S. A., Acosta, L. M., Cobo, J., Nogueras, M. & Palma, A. (2020). *Synthesis*, **52**, 1804–1822.
Nayak, P. S., Narayana, B., Yathirajan, H. S., Hosten, E. C., Betz, R. & Glidewell, C. (2014). *Acta Cryst.* C**70**, 1011–1016.
Omar, W. A. E. & Hormi, O. E. O. (2009). *Tetrahedron*, **65**, 4422–4428.
Polanski, J., Zouhiri, F., Jeanson, L., Desmaële, D., d'Angelo, J., Mouscadet, J. F., Gieleciak, R., Gasteiger, J. & Le Bret, M. (2002). *J. Med. Chem.* **45**, 4647–4654.
Roberts, B. F., Zheng, Y., Cleaveland, J., Lee, S., Lee, E., Ayong, L., Yuan, Y. & Chakrabarti, D. (2017). *Int. J. Parasitol. Drugs Drug Resist.* **7**, 120–129.
Samshuddin, S., Jasinski, J. P., Golen, J. A., Narayana, B., Yathirajan, H. S. & Glidewell, C. (2014). *Acta Cryst.* C**70**, 867–871.
Sharma, R., Kumar, R., Kumar, I. & Sharma, U. (2015). *Eur. J. Org. Chem.* **2015**, 7519–7528.
Sheldrick, G. M. (2015*a*). *Acta Cryst.* A**71**, 3–8.
Sheldrick, G. M. (2015*b*). *Acta Cryst.* C**71**, 3–8.
Spek, A. L. (2015). *Acta Cryst.* C**71**, 9–18.
Spek, A. L. (2020). *Acta Cryst.* E**76**, 1–11.
Valle, G., Busetti, V. & Galiazzo, G. (1986). *Z. Kristallogr.* **177**, 315–318.
Wang, X. Q., Xia, C. L., Chen, S. B., Tan, J. H., Ou, T. M., Huang, S. L., Li, D., Gu, L. Q. & Huang, Z. S. (2015). *Eur. J. Med. Chem.* **89**, 349–361.
Wood, P. A., Allen, F. H. & Pidcock, E. (2009). *CrystEngComm*, **11**, 1563–1571.
Xia, H., Liu, Y., Zhao, P., Gou, S. & Wang, J. (2016). *Org. Lett.* **18**, 1796–1799.

supporting information

Acta Cryst. (2020). C**76** [https://doi.org/10.1107/S2053229620010803]

4-Styrylquinolines from cyclocondensation reactions between (2-aminophenyl)-chalcones and 1,3-diketones: crystal structures and regiochemistry

Diego Rodríguez, Sergio Andrés Guerrero, Alirio Palma, Justo Cobo and Christopher Glidewell

Computing details

For all structures, data collection: *APEX3* (Bruker, 2018); cell refinement: *SAINT* (Bruker, 2017); data reduction: *SAINT* (Bruker, 2017); program(s) used to solve structure: SHELXT2014 (Sheldrick, 2015a); program(s) used to refine structure: *SHELXL2014* (Sheldrick, 2015b); molecular graphics: *PLATON* (Spek, 2020); software used to prepare material for publication: *SHELXL2014* (Sheldrick, 2015b) and *PLATON* (Spek, 2020).

(*E*)-3-Acetyl-4-[2-(4-methoxyphenyl)ethenyl]-2-methylquinoline (I)

Crystal data

$C_{21}H_{19}NO_2$
M_r = 317.37
Monoclinic, $P2_1/c$
a = 8.2595 (4) Å
b = 6.4279 (3) Å
c = 31.9064 (14) Å
β = 93.674 (2)°
V = 1690.47 (14) Å^3
Z = 4
$F(000)$ = 672
D_x = 1.247 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 4198 reflections
θ = 2.5–28.3°
μ = 0.08 mm^{-1}
T = 100 K
Plate, yellow
0.23 × 0.16 × 0.09 mm

Data collection

Bruker D8 Venture diffractometer
Radiation source: INCOATEC high brilliance microfocus sealed tube
Multilayer mirror monochromator
φ and ω scans
Absorption correction: multi-scan (SADABS; Bruker, 2016)
T_{min} = 0.947, T_{max} = 0.993
39800 measured reflections
4197 independent reflections
3528 reflections with $I > 2\sigma(I)$
R_{int} = 0.051
θ_{max} = 28.3°, θ_{min} = 2.6°
h = −11→11
k = −8→8
l = −42→40

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.040
$wR(F^2)$ = 0.111
S = 1.06
4197 reflections
220 parameters
0 restraints
Primary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0488P)^2 + 0.6947P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$

$\Delta\rho_{max} = 0.33$ e Å^{-3}
$\Delta\rho_{min} = -0.23$ e Å^{-3}

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
N1	0.22600 (12)	0.50352 (15)	0.46800 (3)	0.0187 (2)
C2	0.35344 (13)	0.40197 (17)	0.45582 (3)	0.0183 (2)
C3	0.44411 (13)	0.47008 (17)	0.42158 (3)	0.0170 (2)
C4	0.39728 (13)	0.64671 (17)	0.39940 (3)	0.0167 (2)
C4A	0.25816 (13)	0.75745 (17)	0.41187 (3)	0.0170 (2)
C5	0.19665 (15)	0.93792 (19)	0.39100 (4)	0.0220 (2)
H5	0.2493	0.9915	0.3677	0.026*
C6	0.06173 (16)	1.0358 (2)	0.40417 (4)	0.0256 (3)
H6	0.0204	1.1553	0.3896	0.031*
C7	−0.01623 (15)	0.9608 (2)	0.43914 (4)	0.0247 (3)
H7	−0.1088	1.0313	0.4482	0.030*
C8	0.04033 (14)	0.78703 (19)	0.46010 (4)	0.0209 (2)
H8	−0.0127	0.7380	0.4838	0.025*
C8A	0.17774 (13)	0.67992 (17)	0.44663 (3)	0.0171 (2)
C21	0.40445 (16)	0.21170 (19)	0.48070 (4)	0.0238 (2)
H21A	0.3370	0.1977	0.5047	0.036*
H21B	0.5185	0.2251	0.4908	0.036*
H21C	0.3911	0.0883	0.4628	0.036*
C31	0.59731 (14)	0.35584 (18)	0.41244 (3)	0.0203 (2)
O31	0.59378 (12)	0.17673 (14)	0.40028 (3)	0.0326 (2)
C32	0.75319 (14)	0.4693 (2)	0.42241 (4)	0.0245 (3)
H32A	0.7811	0.4627	0.4527	0.037*
H32B	0.7409	0.6150	0.4137	0.037*
H32C	0.8397	0.4045	0.4073	0.037*
C41	0.48647 (13)	0.72134 (18)	0.36375 (3)	0.0185 (2)
H41	0.5124	0.8651	0.3625	0.022*
C42	0.53271 (13)	0.59664 (18)	0.33313 (3)	0.0179 (2)
H42	0.5019	0.4545	0.3345	0.021*
C421	0.62670 (13)	0.65888 (18)	0.29759 (3)	0.0176 (2)
C422	0.70887 (14)	0.84777 (18)	0.29572 (3)	0.0202 (2)
H422	0.7091	0.9402	0.3190	0.024*
C423	0.79067 (14)	0.90422 (19)	0.26062 (4)	0.0211 (2)
H423	0.8458	1.0338	0.2599	0.025*
C424	0.79091 (14)	0.76872 (19)	0.22647 (3)	0.0206 (2)
C425	0.71394 (15)	0.57636 (19)	0.22835 (4)	0.0226 (2)
H425	0.7164	0.4823	0.2054	0.027*

C426	0.63392 (14)	0.52220 (19)	0.26355 (3)	0.0200 (2)
H426	0.5830	0.3900	0.2647	0.024*
O424	0.86368 (11)	0.80822 (15)	0.18996 (3)	0.0275 (2)
C427	0.93382 (16)	1.0093 (2)	0.18527 (4)	0.0274 (3)
H47A	0.8511	1.1163	0.1886	0.041*
H47B	0.9754	1.0211	0.1573	0.041*
H47C	1.0231	1.0286	0.2067	0.041*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.0202 (5)	0.0183 (5)	0.0177 (4)	−0.0008 (4)	0.0010 (3)	0.0008 (4)
C2	0.0204 (5)	0.0159 (5)	0.0182 (5)	−0.0009 (4)	−0.0008 (4)	0.0005 (4)
C3	0.0172 (5)	0.0161 (5)	0.0178 (5)	0.0008 (4)	0.0006 (4)	−0.0020 (4)
C4	0.0185 (5)	0.0163 (5)	0.0154 (5)	0.0009 (4)	0.0019 (4)	−0.0019 (4)
C4A	0.0190 (5)	0.0171 (5)	0.0150 (5)	0.0015 (4)	0.0014 (4)	−0.0015 (4)
C5	0.0280 (6)	0.0223 (6)	0.0162 (5)	0.0056 (5)	0.0057 (4)	0.0025 (4)
C6	0.0292 (6)	0.0251 (6)	0.0231 (6)	0.0098 (5)	0.0044 (5)	0.0044 (5)
C7	0.0219 (6)	0.0271 (6)	0.0257 (6)	0.0075 (5)	0.0059 (4)	0.0000 (5)
C8	0.0194 (5)	0.0246 (6)	0.0192 (5)	0.0002 (4)	0.0044 (4)	−0.0002 (4)
C8A	0.0176 (5)	0.0178 (5)	0.0157 (5)	−0.0005 (4)	0.0006 (4)	−0.0011 (4)
C21	0.0295 (6)	0.0185 (5)	0.0234 (6)	0.0028 (5)	0.0013 (5)	0.0048 (4)
C31	0.0237 (6)	0.0188 (5)	0.0185 (5)	0.0046 (4)	0.0016 (4)	0.0004 (4)
O31	0.0335 (5)	0.0205 (4)	0.0437 (6)	0.0063 (4)	0.0013 (4)	−0.0083 (4)
C32	0.0204 (6)	0.0273 (6)	0.0259 (6)	0.0038 (5)	0.0030 (4)	−0.0002 (5)
C41	0.0201 (5)	0.0176 (5)	0.0180 (5)	0.0019 (4)	0.0030 (4)	0.0013 (4)
C42	0.0176 (5)	0.0182 (5)	0.0178 (5)	0.0009 (4)	0.0009 (4)	−0.0001 (4)
C421	0.0166 (5)	0.0197 (5)	0.0164 (5)	0.0025 (4)	0.0015 (4)	−0.0011 (4)
C422	0.0205 (5)	0.0215 (6)	0.0188 (5)	0.0011 (4)	0.0030 (4)	−0.0044 (4)
C423	0.0205 (5)	0.0211 (5)	0.0220 (5)	−0.0023 (4)	0.0032 (4)	−0.0025 (4)
C424	0.0194 (5)	0.0256 (6)	0.0173 (5)	0.0007 (4)	0.0042 (4)	−0.0003 (4)
C425	0.0264 (6)	0.0244 (6)	0.0173 (5)	−0.0020 (5)	0.0043 (4)	−0.0055 (4)
C426	0.0206 (5)	0.0205 (5)	0.0189 (5)	−0.0010 (4)	0.0018 (4)	−0.0029 (4)
O424	0.0341 (5)	0.0297 (5)	0.0199 (4)	−0.0070 (4)	0.0109 (3)	−0.0027 (3)
C427	0.0276 (6)	0.0314 (7)	0.0240 (6)	−0.0065 (5)	0.0066 (5)	0.0027 (5)

Geometric parameters (Å, º)

N1—C2	1.3181 (15)	C32—H32A	0.9800
N1—C8A	1.3692 (14)	C32—H32B	0.9800
C2—C3	1.4319 (15)	C32—H32C	0.9800
C2—C21	1.5036 (16)	C41—C42	1.3382 (15)
C3—C4	1.3800 (15)	C41—H41	0.9500
C3—C31	1.5076 (15)	C42—C421	1.4701 (15)
C4—C4A	1.4296 (15)	C42—H42	0.9500
C4—C41	1.4746 (15)	C421—C422	1.3941 (16)
C4A—C5	1.4157 (16)	C421—C426	1.4012 (15)
C4A—C8A	1.4190 (15)	C422—C423	1.3926 (16)

C5—C6	1.3691 (16)	C422—H422	0.9500
C5—H5	0.9500	C423—C424	1.3952 (16)
C6—C7	1.4087 (17)	C423—H423	0.9500
C6—H6	0.9500	C424—O424	1.3684 (13)
C7—C8	1.3685 (17)	C424—C425	1.3934 (17)
C7—H7	0.9500	C425—C426	1.3837 (15)
C8—C8A	1.4176 (15)	C425—H425	0.9500
C8—H8	0.9500	C426—H426	0.9500
C21—H21A	0.9800	O424—C427	1.4282 (15)
C21—H21B	0.9800	C427—H47A	0.9800
C21—H21C	0.9800	C427—H47B	0.9800
C31—O31	1.2147 (15)	C427—H47C	0.9800
C31—C32	1.4959 (17)		

C2—N1—C8A	118.41 (9)	C31—C32—H32A	109.5
N1—C2—C3	122.69 (10)	C31—C32—H32B	109.5
N1—C2—C21	116.69 (10)	H32A—C32—H32B	109.5
C3—C2—C21	120.59 (10)	C31—C32—H32C	109.5
C4—C3—C2	119.97 (10)	H32A—C32—H32C	109.5
C4—C3—C31	120.84 (10)	H32B—C32—H32C	109.5
C2—C3—C31	119.03 (10)	C42—C41—C4	123.26 (11)
C3—C4—C4A	118.09 (10)	C42—C41—H41	118.4
C3—C4—C41	121.72 (10)	C4—C41—H41	118.4
C4A—C4—C41	120.19 (10)	C41—C42—C421	126.13 (11)
C5—C4A—C8A	118.92 (10)	C41—C42—H42	116.9
C5—C4A—C4	123.21 (10)	C421—C42—H42	116.9
C8A—C4A—C4	117.87 (10)	C422—C421—C426	117.90 (10)
C6—C5—C4A	120.45 (11)	C422—C421—C42	123.54 (10)
C6—C5—H5	119.8	C426—C421—C42	118.56 (10)
C4A—C5—H5	119.8	C423—C422—C421	121.59 (10)
C5—C6—C7	120.54 (11)	C423—C422—H422	119.2
C5—C6—H6	119.7	C421—C422—H422	119.2
C7—C6—H6	119.7	C422—C423—C424	119.38 (11)
C8—C7—C6	120.49 (11)	C422—C423—H423	120.3
C8—C7—H7	119.8	C424—C423—H423	120.3
C6—C7—H7	119.8	O424—C424—C425	115.33 (10)
C7—C8—C8A	120.20 (10)	O424—C424—C423	124.88 (11)
C7—C8—H8	119.9	C425—C424—C423	119.79 (10)
C8A—C8—H8	119.9	C426—C425—C424	120.08 (11)
N1—C8A—C8	117.67 (10)	C426—C425—H425	120.0
N1—C8A—C4A	122.96 (10)	C424—C425—H425	120.0
C8—C8A—C4A	119.37 (10)	C425—C426—C421	121.18 (11)
C2—C21—H21A	109.5	C425—C426—H426	119.4
C2—C21—H21B	109.5	C421—C426—H426	119.4
H21A—C21—H21B	109.5	C424—O424—C427	117.40 (9)
C2—C21—H21C	109.5	O424—C427—H47A	109.5
H21A—C21—H21C	109.5	O424—C427—H47B	109.5
H21B—C21—H21C	109.5	H47A—C427—H47B	109.5

O31—C31—C32	122.18 (11)	O424—C427—H47C	109.5
O31—C31—C3	121.35 (11)	H47A—C427—H47C	109.5
C32—C31—C3	116.30 (10)	H47B—C427—H47C	109.5
C8A—N1—C2—C3	0.77 (16)	C4—C4A—C8A—N1	−1.07 (16)
C8A—N1—C2—C21	178.74 (10)	C5—C4A—C8A—C8	−1.30 (16)
N1—C2—C3—C4	−1.17 (17)	C4—C4A—C8A—C8	179.38 (10)
C21—C2—C3—C4	−179.06 (10)	C4—C3—C31—O31	−118.43 (13)
N1—C2—C3—C31	174.26 (10)	C2—C3—C31—O31	66.18 (15)
C21—C2—C3—C31	−3.63 (16)	C4—C3—C31—C32	66.26 (14)
C2—C3—C4—C4A	0.39 (16)	C2—C3—C31—C32	−109.13 (12)
C31—C3—C4—C4A	−174.96 (10)	C3—C4—C41—C42	46.40 (16)
C2—C3—C4—C41	−179.53 (10)	C4A—C4—C41—C42	−133.52 (12)
C31—C3—C4—C41	5.12 (16)	C4—C41—C42—C421	−177.66 (10)
C3—C4—C4A—C5	−178.64 (11)	C41—C42—C421—C422	13.78 (18)
C41—C4—C4A—C5	1.29 (17)	C41—C42—C421—C426	−165.87 (11)
C3—C4—C4A—C8A	0.65 (15)	C426—C421—C422—C423	2.48 (17)
C41—C4—C4A—C8A	−179.43 (10)	C42—C421—C422—C423	−177.17 (11)
C8A—C4A—C5—C6	−0.01 (18)	C421—C422—C423—C424	−0.12 (18)
C4—C4A—C5—C6	179.27 (12)	C422—C423—C424—O424	178.54 (11)
C4A—C5—C6—C7	1.2 (2)	C422—C423—C424—C425	−1.99 (18)
C5—C6—C7—C8	−1.0 (2)	O424—C424—C425—C426	−178.81 (11)
C6—C7—C8—C8A	−0.36 (19)	C423—C424—C425—C426	1.68 (18)
C2—N1—C8A—C8	179.91 (10)	C424—C425—C426—C421	0.77 (18)
C2—N1—C8A—C4A	0.36 (16)	C422—C421—C426—C425	−2.80 (17)
C7—C8—C8A—N1	−178.08 (11)	C42—C421—C426—C425	176.87 (11)
C7—C8—C8A—C4A	1.49 (17)	C425—C424—O424—C427	175.49 (11)
C5—C4A—C8A—N1	178.24 (10)	C423—C424—O424—C427	−5.02 (17)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C41—H41···O31^{i}	0.95	2.41	3.2527 (15)	148
C426—H426···*Cg*3^{ii}	0.95	2.77	3.5252 (13)	138

Symmetry codes: (i) *x*, *y*+1, *z*; (ii) −*x*+1, *y*−1/2, −*z*+1/2.

(*E*)-3-Acetyl-4-[2-(4-bromophenyl)ethenyl]-2-methylquinoline (II)

Crystal data

$C_{20}H_{16}BrNO$
M_r = 366.24
Monoclinic, $P2_1/c$
a = 8.0849 (3) Å
b = 6.6692 (2) Å
c = 31.1063 (10) Å
β = 95.005 (1)°
V = 1670.85 (10) Å^3
Z = 4
$F(000)$ = 744
D_x = 1.456 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 3839 reflections
θ = 2.5–27.5°
μ = 2.46 mm^{-1}
T = 100 K
Plate, yellow
0.17 × 0.11 × 0.04 mm

Data collection

Bruker D8 Venture diffractometer
Radiation source: INCOATEC high brilliance microfocus sealed tube
Multilayer mirror monochromator
φ and ω scans
Absorption correction: multi-scan (SADABS; Bruker, 2016)
T_{min} = 0.810, T_{max} = 0.906
38342 measured reflections
3839 independent reflections
3434 reflections with $I > 2\sigma(I)$
R_{int} = 0.034
θ_{max} = 27.5°, θ_{min} = 2.5°
h = −10→10
k = −8→8
l = −39→40

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.030
$wR(F^2)$ = 0.073
S = 1.03
3839 reflections
210 parameters
0 restraints
Primary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0294P)^2 + 1.5332P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.56 e Å^{-3}
$\Delta\rho_{min}$ = −0.76 e Å^{-3}

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	y	z	U_{iso}*/U_{eq}
N1	0.22878 (18)	0.4980 (2)	0.46764 (5)	0.0216 (3)
C2	0.3566 (2)	0.4009 (2)	0.45402 (5)	0.0214 (3)
C3	0.4453 (2)	0.4719 (2)	0.41918 (5)	0.0198 (3)
C4	0.3970 (2)	0.6464 (2)	0.39805 (5)	0.0198 (3)
C4A	0.2553 (2)	0.7499 (2)	0.41163 (5)	0.0211 (3)
C5	0.1897 (2)	0.9251 (3)	0.39104 (6)	0.0314 (4)
H5	0.2403	0.9788	0.3671	0.038*
C6	0.0535 (3)	1.0184 (3)	0.40534 (7)	0.0358 (5)
H6	0.0093	1.1347	0.3909	0.043*
C7	−0.0209 (2)	0.9429 (3)	0.44112 (6)	0.0302 (4)
H7	−0.1138	1.0101	0.4511	0.036*
C8	0.0395 (2)	0.7731 (3)	0.46174 (6)	0.0249 (3)
H8	−0.0110	0.7237	0.4861	0.030*
C8A	0.1770 (2)	0.6713 (2)	0.44684 (5)	0.0203 (3)
C21	0.4107 (2)	0.2140 (3)	0.47846 (6)	0.0290 (4)
H21A	0.3457	0.1989	0.5034	0.043*
H21B	0.5288	0.2242	0.4884	0.043*
H21C	0.3927	0.0972	0.4595	0.043*
C31	0.5978 (2)	0.3597 (3)	0.40803 (6)	0.0248 (3)

O31	0.58723 (19)	0.1923 (2)	0.39267 (5)	0.0402 (3)
C32	0.7612 (2)	0.4597 (3)	0.41940 (7)	0.0315 (4)
H32A	0.7915	0.4466	0.4505	0.047*
H32B	0.7527	0.6021	0.4117	0.047*
H32C	0.8465	0.3961	0.4035	0.047*
C41	0.4876 (2)	0.7319 (3)	0.36336 (5)	0.0221 (3)
H41	0.5115	0.8713	0.3646	0.026*
C42	0.5388 (2)	0.6286 (3)	0.33029 (5)	0.0218 (3)
H42	0.5101	0.4906	0.3282	0.026*
C421	0.63654 (19)	0.7121 (3)	0.29676 (5)	0.0223 (3)
C422	0.7176 (2)	0.8969 (3)	0.30115 (6)	0.0263 (4)
H422	0.7123	0.9718	0.3270	0.032*
C423	0.8056 (2)	0.9731 (3)	0.26847 (6)	0.0298 (4)
H423	0.8603	1.0989	0.2717	0.036*
C424	0.8123 (2)	0.8621 (3)	0.23090 (6)	0.0303 (4)
Br42	0.93043 (2)	0.96822 (4)	0.18577 (2)	0.04244 (9)
C425	0.7361 (2)	0.6784 (4)	0.22581 (6)	0.0368 (5)
H425	0.7429	0.6037	0.2000	0.044*
C426	0.6488 (2)	0.6033 (3)	0.25888 (6)	0.0304 (4)
H426	0.5967	0.4758	0.2556	0.037*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.0235 (7)	0.0197 (7)	0.0218 (7)	−0.0007 (5)	0.0040 (5)	0.0022 (5)
C2	0.0252 (8)	0.0163 (7)	0.0228 (8)	−0.0003 (6)	0.0019 (6)	0.0013 (6)
C3	0.0221 (7)	0.0167 (7)	0.0209 (8)	0.0011 (6)	0.0025 (6)	−0.0029 (6)
C4	0.0234 (7)	0.0181 (7)	0.0183 (7)	0.0017 (6)	0.0035 (6)	−0.0014 (6)
C4A	0.0248 (8)	0.0206 (8)	0.0183 (7)	0.0039 (6)	0.0037 (6)	0.0008 (6)
C5	0.0389 (10)	0.0314 (10)	0.0257 (9)	0.0141 (8)	0.0128 (7)	0.0104 (7)
C6	0.0420 (11)	0.0338 (10)	0.0329 (10)	0.0195 (9)	0.0106 (8)	0.0112 (8)
C7	0.0282 (9)	0.0318 (10)	0.0317 (9)	0.0111 (7)	0.0095 (7)	0.0020 (8)
C8	0.0243 (8)	0.0280 (9)	0.0231 (8)	0.0017 (7)	0.0068 (6)	0.0004 (7)
C8A	0.0220 (7)	0.0196 (8)	0.0193 (7)	0.0005 (6)	0.0024 (6)	0.0001 (6)
C21	0.0359 (9)	0.0205 (8)	0.0312 (9)	0.0040 (7)	0.0066 (7)	0.0076 (7)
C31	0.0309 (9)	0.0193 (8)	0.0250 (8)	0.0067 (7)	0.0076 (7)	0.0026 (6)
O31	0.0482 (8)	0.0202 (7)	0.0543 (9)	0.0054 (6)	0.0170 (7)	−0.0086 (6)
C32	0.0252 (9)	0.0346 (10)	0.0351 (10)	0.0085 (7)	0.0041 (7)	0.0015 (8)
C41	0.0259 (8)	0.0195 (8)	0.0215 (8)	0.0035 (6)	0.0058 (6)	0.0022 (6)
C42	0.0205 (7)	0.0239 (8)	0.0212 (8)	0.0009 (6)	0.0025 (6)	−0.0013 (6)
C421	0.0182 (7)	0.0305 (9)	0.0184 (8)	0.0026 (6)	0.0026 (6)	−0.0014 (6)
C422	0.0247 (8)	0.0319 (9)	0.0227 (8)	0.0003 (7)	0.0046 (6)	−0.0039 (7)
C423	0.0253 (8)	0.0352 (10)	0.0295 (9)	−0.0019 (7)	0.0059 (7)	0.0019 (8)
C424	0.0198 (8)	0.0518 (12)	0.0200 (8)	0.0001 (8)	0.0054 (6)	0.0042 (8)
Br42	0.03101 (11)	0.07124 (17)	0.02652 (11)	−0.00315 (9)	0.01070 (8)	0.01007 (9)
C425	0.0309 (9)	0.0599 (14)	0.0205 (9)	−0.0071 (9)	0.0063 (7)	−0.0119 (9)
C426	0.0267 (8)	0.0405 (11)	0.0246 (9)	−0.0067 (8)	0.0053 (7)	−0.0100 (8)

Geometric parameters (Å, º)

N1—C2	1.321 (2)	C31—O31	1.214 (2)
N1—C8A	1.372 (2)	C31—C32	1.495 (3)
C2—C3	1.431 (2)	C32—H32A	0.9800
C2—C21	1.505 (2)	C32—H32B	0.9800
C3—C4	1.377 (2)	C32—H32C	0.9800
C3—C31	1.509 (2)	C41—C42	1.334 (2)
C4—C4A	1.432 (2)	C41—H41	0.9500
C4—C41	1.471 (2)	C42—C421	1.472 (2)
C4A—C8A	1.413 (2)	C42—H42	0.9500
C4A—C5	1.413 (2)	C421—C426	1.395 (2)
C5—C6	1.372 (3)	C421—C422	1.397 (3)
C5—H5	0.9500	C422—C423	1.387 (3)
C6—C7	1.404 (3)	C422—H422	0.9500
C6—H6	0.9500	C423—C424	1.388 (3)
C7—C8	1.371 (3)	C423—H423	0.9500
C7—H7	0.9500	C424—C425	1.375 (3)
C8—C8A	1.415 (2)	C424—Br42	1.9018 (17)
C8—H8	0.9500	C425—C426	1.390 (3)
C21—H21A	0.9800	C425—H425	0.9500
C21—H21B	0.9800	C426—H426	0.9500
C21—H21C	0.9800		
C2—N1—C8A	118.51 (14)	O31—C31—C32	122.29 (16)
N1—C2—C3	122.57 (15)	O31—C31—C3	121.02 (17)
N1—C2—C21	116.44 (15)	C32—C31—C3	116.59 (15)
C3—C2—C21	120.94 (15)	C31—C32—H32A	109.5
C4—C3—C2	120.10 (15)	C31—C32—H32B	109.5
C4—C3—C31	120.88 (15)	H32A—C32—H32B	109.5
C2—C3—C31	118.92 (14)	C31—C32—H32C	109.5
C3—C4—C4A	117.88 (15)	H32A—C32—H32C	109.5
C3—C4—C41	122.63 (14)	H32B—C32—H32C	109.5
C4A—C4—C41	119.47 (14)	C42—C41—C4	125.02 (16)
C8A—C4A—C5	118.84 (15)	C42—C41—H41	117.5
C8A—C4A—C4	118.27 (15)	C4—C41—H41	117.5
C5—C4A—C4	122.89 (15)	C41—C42—C421	124.98 (16)
C6—C5—C4A	120.53 (17)	C41—C42—H42	117.5
C6—C5—H5	119.7	C421—C42—H42	117.5
C4A—C5—H5	119.7	C426—C421—C422	118.32 (16)
C5—C6—C7	120.41 (17)	C426—C421—C42	118.94 (16)
C5—C6—H6	119.8	C422—C421—C42	122.74 (15)
C7—C6—H6	119.8	C423—C422—C421	121.18 (17)
C8—C7—C6	120.49 (16)	C423—C422—H422	119.4
C8—C7—H7	119.8	C421—C422—H422	119.4
C6—C7—H7	119.8	C422—C423—C424	118.80 (18)
C7—C8—C8A	120.09 (16)	C422—C423—H423	120.6
C7—C8—H8	120.0	C424—C423—H423	120.6

C8A—C8—H8	120.0	C425—C424—C423	121.48 (17)
N1—C8A—C4A	122.63 (15)	C425—C424—Br42	119.71 (14)
N1—C8A—C8	117.77 (15)	C423—C424—Br42	118.81 (15)
C4A—C8A—C8	119.59 (15)	C424—C425—C426	119.16 (17)
C2—C21—H21A	109.5	C424—C425—H425	120.4
C2—C21—H21B	109.5	C426—C425—H425	120.4
H21A—C21—H21B	109.5	C425—C426—C421	121.04 (18)
C2—C21—H21C	109.5	C425—C426—H426	119.5
H21A—C21—H21C	109.5	C421—C426—H426	119.5
H21B—C21—H21C	109.5		

C8A—N1—C2—C3	1.5 (2)	C5—C4A—C8A—C8	−2.4 (2)
C8A—N1—C2—C21	178.81 (15)	C4—C4A—C8A—C8	178.33 (15)
N1—C2—C3—C4	−0.9 (3)	C7—C8—C8A—N1	−177.65 (16)
C21—C2—C3—C4	−178.04 (16)	C7—C8—C8A—C4A	2.3 (3)
N1—C2—C3—C31	175.33 (15)	C4—C3—C31—O31	−115.6 (2)
C21—C2—C3—C31	−1.8 (2)	C2—C3—C31—O31	68.2 (2)
C2—C3—C4—C4A	−1.1 (2)	C4—C3—C31—C32	68.1 (2)
C31—C3—C4—C4A	−177.20 (15)	C2—C3—C31—C32	−108.09 (18)
C2—C3—C4—C41	177.13 (15)	C3—C4—C41—C42	46.8 (3)
C31—C3—C4—C41	1.0 (2)	C4A—C4—C41—C42	−135.02 (18)
C3—C4—C4A—C8A	2.2 (2)	C4—C41—C42—C421	−176.86 (15)
C41—C4—C4A—C8A	−176.01 (15)	C41—C42—C421—C426	−165.04 (17)
C3—C4—C4A—C5	−176.96 (17)	C41—C42—C421—C422	14.8 (3)
C41—C4—C4A—C5	4.8 (3)	C426—C421—C422—C423	1.2 (3)
C8A—C4A—C5—C6	0.7 (3)	C42—C421—C422—C423	−178.57 (16)
C4—C4A—C5—C6	179.93 (19)	C421—C422—C423—C424	0.0 (3)
C4A—C5—C6—C7	1.1 (3)	C422—C423—C424—C425	−1.0 (3)
C5—C6—C7—C8	−1.2 (3)	C422—C423—C424—Br42	179.13 (14)
C6—C7—C8—C8A	−0.5 (3)	C423—C424—C425—C426	0.7 (3)
C2—N1—C8A—C4A	−0.2 (2)	Br42—C424—C425—C426	−179.40 (15)
C2—N1—C8A—C8	179.77 (15)	C424—C425—C426—C421	0.6 (3)
C5—C4A—C8A—N1	177.56 (16)	C422—C421—C426—C425	−1.5 (3)
C4—C4A—C8A—N1	−1.7 (2)	C42—C421—C426—C425	178.30 (17)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C41—H41···O31^{i}	0.95	2.37	3.283 (2)	161
C426—H426···*Cg*3^{ii}	0.95	2.91	3.7071 (19)	142

Symmetry codes: (i) *x*, *y*+1, *z*; (ii) −*x*+1, *y*−1/2, −*z*+1/2.

(*E*)-3-Acetyl-2-methyl-4-{2-[4-(trifluoromethyl)phenyl]ethenyl}quinoline (III)

Crystal data

$C_{21}H_{16}F_3NO$	*a* = 8.0822 (4) Å
M_r = 355.35	*b* = 6.6567 (4) Å
Monoclinic, $P2_1/c$	*c* = 32.1024 (17) Å

β = 90.576 (2)°
V = 1727.05 (16) Å^3
Z = 4
F(000) = 736
D_x = 1.367 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å

Cell parameters from 3961 reflections
θ = 2.5–27.5°
μ = 0.11 mm^{-1}
T = 100 K
Needle, colourless
0.20 × 0.08 × 0.06 mm

Data collection

Bruker D8 Venture diffractometer
Radiation source: INCOATEC high brilliance microfocus sealed tube
Multilayer mirror monochromator
φ and ω scans
Absorption correction: multi-scan (SADABS; Bruker, 2016)
T_{min} = 0.942, T_{max} = 0.994

19648 measured reflections
3959 independent reflections
3363 reflections with $I > 2\sigma(I)$
R_{int} = 0.032
θ_{max} = 27.5°, θ_{min} = 2.5°
h = −10→10
k = −8→8
l = −41→40

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.041
$wR(F^2)$ = 0.101
S = 1.04
3959 reflections
237 parameters
0 restraints

Primary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.039P)^2 + 0.8719P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.28 e Å^{-3}
$\Delta\rho_{min}$ = −0.23 e Å^{-3}

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	y	z	U_{iso}*/U_{eq}
N1	0.24277 (14)	0.49703 (17)	0.46953 (3)	0.0222 (2)
C2	0.37353 (17)	0.40329 (19)	0.45511 (4)	0.0214 (3)
C3	0.46663 (16)	0.47574 (19)	0.42048 (4)	0.0198 (3)
C4	0.41905 (16)	0.65014 (19)	0.40063 (4)	0.0200 (3)
C4A	0.27453 (17)	0.7513 (2)	0.41515 (4)	0.0213 (3)
C5	0.2095 (2)	0.9254 (2)	0.39603 (5)	0.0312 (3)
H5	0.2639	0.9819	0.3727	0.037*
C6	0.0690 (2)	1.0136 (3)	0.41073 (5)	0.0382 (4)
H6	0.0251	1.1291	0.3972	0.046*
C7	−0.01102 (19)	0.9344 (2)	0.44580 (5)	0.0334 (3)
H7	−0.1075	0.9982	0.4561	0.040*
C8	0.04868 (17)	0.7666 (2)	0.46520 (4)	0.0263 (3)
H8	−0.0056	0.7150	0.4890	0.032*

C8A	0.19136 (16)	0.6693 (2)	0.44989 (4)	0.0207 (3)
C21	0.42652 (19)	0.2158 (2)	0.47786 (5)	0.0287 (3)
H21A	0.3609	0.2007	0.5032	0.043*
H21B	0.5440	0.2258	0.4854	0.043*
H21C	0.4091	0.0988	0.4598	0.043*
C31	0.62124 (18)	0.3660 (2)	0.40773 (4)	0.0248 (3)
O31	0.61447 (15)	0.20068 (16)	0.39209 (4)	0.0390 (3)
C32	0.78127 (18)	0.4677 (2)	0.41812 (5)	0.0320 (3)
H32A	0.8021	0.4591	0.4482	0.048*
H32B	0.7752	0.6092	0.4098	0.048*
H32C	0.8714	0.4016	0.4032	0.048*
C41	0.50967 (16)	0.7346 (2)	0.36505 (4)	0.0218 (3)
H41	0.5375	0.8732	0.3659	0.026*
C42	0.55522 (16)	0.6296 (2)	0.33171 (4)	0.0207 (3)
H42	0.5279	0.4908	0.3311	0.025*
C421	0.64460 (15)	0.7119 (2)	0.29577 (4)	0.0203 (3)
C422	0.72004 (17)	0.9002 (2)	0.29629 (4)	0.0245 (3)
H422	0.7169	0.9796	0.3209	0.029*
C423	0.79950 (17)	0.9730 (2)	0.26140 (4)	0.0258 (3)
H423	0.8496	1.1021	0.2621	0.031*
C424	0.80597 (16)	0.8576 (2)	0.22548 (4)	0.0236 (3)
C425	0.73412 (18)	0.6685 (2)	0.22450 (4)	0.0280 (3)
H425	0.7395	0.5887	0.2000	0.034*
C426	0.65429 (17)	0.5967 (2)	0.25951 (4)	0.0255 (3)
H426	0.6054	0.4669	0.2588	0.031*
C427	0.89461 (18)	0.9343 (2)	0.18789 (4)	0.0284 (3)
F471	1.04888 (11)	0.85971 (18)	0.18484 (3)	0.0461 (3)
F472	0.90958 (14)	1.13364 (15)	0.18753 (3)	0.0461 (3)
F473	0.81762 (11)	0.88303 (14)	0.15231 (2)	0.0328 (2)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.0276 (6)	0.0200 (6)	0.0190 (5)	−0.0029 (4)	0.0008 (4)	0.0013 (4)
C2	0.0292 (7)	0.0162 (6)	0.0187 (6)	−0.0027 (5)	−0.0015 (5)	0.0002 (5)
C3	0.0263 (6)	0.0155 (6)	0.0177 (6)	−0.0003 (5)	0.0004 (5)	−0.0025 (5)
C4	0.0265 (6)	0.0176 (6)	0.0161 (6)	0.0001 (5)	0.0010 (5)	−0.0021 (5)
C4A	0.0284 (7)	0.0190 (6)	0.0165 (6)	0.0021 (5)	0.0020 (5)	−0.0003 (5)
C5	0.0418 (8)	0.0288 (8)	0.0234 (7)	0.0117 (6)	0.0097 (6)	0.0084 (6)
C6	0.0478 (9)	0.0338 (9)	0.0331 (8)	0.0201 (7)	0.0104 (7)	0.0105 (7)
C7	0.0337 (8)	0.0350 (8)	0.0315 (8)	0.0112 (7)	0.0099 (6)	0.0015 (7)
C8	0.0280 (7)	0.0284 (7)	0.0226 (7)	0.0001 (6)	0.0058 (5)	0.0013 (6)
C8A	0.0253 (6)	0.0202 (6)	0.0166 (6)	−0.0006 (5)	0.0001 (5)	−0.0006 (5)
C21	0.0404 (8)	0.0206 (7)	0.0250 (7)	0.0020 (6)	0.0012 (6)	0.0056 (6)
C31	0.0356 (7)	0.0189 (7)	0.0199 (6)	0.0066 (6)	0.0038 (5)	0.0025 (5)
O31	0.0541 (7)	0.0205 (5)	0.0426 (7)	0.0076 (5)	0.0088 (5)	−0.0074 (5)
C32	0.0281 (7)	0.0367 (8)	0.0311 (8)	0.0088 (6)	0.0020 (6)	0.0001 (7)
C41	0.0267 (6)	0.0177 (6)	0.0210 (6)	0.0024 (5)	0.0029 (5)	0.0013 (5)

C42	0.0225 (6)	0.0198 (6)	0.0200 (6)	−0.0003 (5)	0.0007 (5)	0.0004 (5)
C421	0.0191 (6)	0.0234 (7)	0.0185 (6)	0.0024 (5)	0.0006 (5)	−0.0004 (5)
C422	0.0270 (7)	0.0262 (7)	0.0202 (7)	−0.0020 (5)	0.0031 (5)	−0.0047 (5)
C423	0.0265 (7)	0.0253 (7)	0.0256 (7)	−0.0042 (5)	0.0023 (5)	−0.0005 (6)
C424	0.0215 (6)	0.0302 (7)	0.0193 (6)	−0.0008 (5)	0.0015 (5)	0.0024 (6)
C425	0.0333 (7)	0.0326 (8)	0.0182 (7)	−0.0035 (6)	0.0038 (5)	−0.0052 (6)
C426	0.0293 (7)	0.0250 (7)	0.0224 (7)	−0.0039 (6)	0.0033 (5)	−0.0031 (6)
C427	0.0276 (7)	0.0349 (8)	0.0226 (7)	−0.0008 (6)	0.0027 (5)	0.0038 (6)
F471	0.0250 (4)	0.0760 (8)	0.0377 (5)	0.0060 (5)	0.0088 (4)	0.0219 (5)
F472	0.0679 (7)	0.0365 (5)	0.0341 (5)	−0.0151 (5)	0.0131 (5)	0.0051 (4)
F473	0.0361 (5)	0.0443 (5)	0.0182 (4)	0.0004 (4)	0.0018 (3)	0.0027 (4)

Geometric parameters (Å, º)

N1—C2	1.3154 (18)	C31—C32	1.495 (2)
N1—C8A	1.3710 (17)	C32—H32A	0.9800
C2—C3	1.4324 (18)	C32—H32B	0.9800
C2—C21	1.5062 (18)	C32—H32C	0.9800
C3—C4	1.3772 (18)	C41—C42	1.3335 (18)
C3—C31	1.5077 (18)	C41—H41	0.9500
C4—C4A	1.4304 (18)	C42—C421	1.4729 (18)
C4—C41	1.4746 (18)	C42—H42	0.9500
C4A—C5	1.4102 (19)	C421—C422	1.3942 (19)
C4A—C8A	1.4175 (18)	C421—C426	1.3969 (19)
C5—C6	1.367 (2)	C422—C423	1.3843 (19)
C5—H5	0.9500	C422—H422	0.9500
C6—C7	1.407 (2)	C423—C424	1.387 (2)
C6—H6	0.9500	C423—H423	0.9500
C7—C8	1.365 (2)	C424—C425	1.386 (2)
C7—H7	0.9500	C424—C427	1.4991 (19)
C8—C8A	1.4151 (19)	C425—C426	1.3866 (19)
C8—H8	0.9500	C425—H425	0.9500
C21—H21A	0.9800	C426—H426	0.9500
C21—H21B	0.9800	C427—F472	1.3326 (18)
C21—H21C	0.9800	C427—F473	1.3392 (17)
C31—O31	1.2105 (18)	C427—F471	1.3465 (17)
C2—N1—C8A	118.41 (11)	C31—C32—H32A	109.5
N1—C2—C3	122.93 (12)	C31—C32—H32B	109.5
N1—C2—C21	116.62 (12)	H32A—C32—H32B	109.5
C3—C2—C21	120.42 (12)	C31—C32—H32C	109.5
C4—C3—C2	119.77 (12)	H32A—C32—H32C	109.5
C4—C3—C31	120.76 (12)	H32B—C32—H32C	109.5
C2—C3—C31	119.39 (12)	C42—C41—C4	124.41 (12)
C3—C4—C4A	118.13 (12)	C42—C41—H41	117.8
C3—C4—C41	122.77 (12)	C4—C41—H41	117.8
C4A—C4—C41	119.10 (12)	C41—C42—C421	125.11 (13)
C5—C4A—C8A	118.78 (12)	C41—C42—H42	117.4

C5—C4A—C4	123.17 (12)	C421—C42—H42	117.4
C8A—C4A—C4	118.04 (12)	C422—C421—C426	118.39 (12)
C6—C5—C4A	120.67 (14)	C422—C421—C42	122.90 (12)
C6—C5—H5	119.7	C426—C421—C42	118.72 (12)
C4A—C5—H5	119.7	C423—C422—C421	120.76 (13)
C5—C6—C7	120.36 (14)	C423—C422—H422	119.6
C5—C6—H6	119.8	C421—C422—H422	119.6
C7—C6—H6	119.8	C422—C423—C424	120.03 (13)
C8—C7—C6	120.60 (14)	C422—C423—H423	120.0
C8—C7—H7	119.7	C424—C423—H423	120.0
C6—C7—H7	119.7	C425—C424—C423	120.15 (13)
C7—C8—C8A	120.11 (13)	C425—C424—C427	119.64 (13)
C7—C8—H8	119.9	C423—C424—C427	120.19 (13)
C8A—C8—H8	119.9	C424—C425—C426	119.55 (13)
N1—C8A—C8	117.86 (12)	C424—C425—H425	120.2
N1—C8A—C4A	122.69 (12)	C426—C425—H425	120.2
C8—C8A—C4A	119.44 (12)	C425—C426—C421	121.10 (13)
C2—C21—H21A	109.5	C425—C426—H426	119.5
C2—C21—H21B	109.5	C421—C426—H426	119.5
H21A—C21—H21B	109.5	F472—C427—F473	106.72 (12)
C2—C21—H21C	109.5	F472—C427—F471	106.39 (13)
H21A—C21—H21C	109.5	F473—C427—F471	105.47 (12)
H21B—C21—H21C	109.5	F472—C427—C424	112.97 (13)
O31—C31—C32	122.68 (14)	F473—C427—C424	112.21 (12)
O31—C31—C3	121.26 (14)	F471—C427—C424	112.54 (12)
C32—C31—C3	115.94 (12)		

C8A—N1—C2—C3	1.51 (19)	C4—C4A—C8A—C8	179.55 (12)
C8A—N1—C2—C21	179.47 (12)	C4—C3—C31—O31	−113.91 (16)
N1—C2—C3—C4	−0.5 (2)	C2—C3—C31—O31	69.52 (18)
C21—C2—C3—C4	−178.38 (12)	C4—C3—C31—C32	69.90 (17)
N1—C2—C3—C31	176.10 (12)	C2—C3—C31—C32	−106.67 (15)
C21—C2—C3—C31	−1.78 (19)	C3—C4—C41—C42	49.0 (2)
C2—C3—C4—C4A	−1.18 (19)	C4A—C4—C41—C42	−130.33 (14)
C31—C3—C4—C4A	−177.74 (12)	C4—C41—C42—C421	179.51 (12)
C2—C3—C4—C41	179.44 (12)	C41—C42—C421—C422	13.0 (2)
C31—C3—C4—C41	2.9 (2)	C41—C42—C421—C426	−166.89 (13)
C3—C4—C4A—C5	−176.83 (13)	C426—C421—C422—C423	1.4 (2)
C41—C4—C4A—C5	2.6 (2)	C42—C421—C422—C423	−178.54 (13)
C3—C4—C4A—C8A	1.77 (19)	C421—C422—C423—C424	−0.5 (2)
C41—C4—C4A—C8A	−178.83 (12)	C422—C423—C424—C425	−0.5 (2)
C8A—C4A—C5—C6	0.1 (2)	C422—C423—C424—C427	−178.86 (13)
C4—C4A—C5—C6	178.67 (15)	C423—C424—C425—C426	0.7 (2)
C4A—C5—C6—C7	1.4 (3)	C427—C424—C425—C426	179.06 (13)
C5—C6—C7—C8	−1.1 (3)	C424—C425—C426—C421	0.2 (2)
C6—C7—C8—C8A	−0.6 (2)	C422—C421—C426—C425	−1.2 (2)
C2—N1—C8A—C8	178.81 (12)	C42—C421—C426—C425	178.73 (13)
C2—N1—C8A—C4A	−0.84 (19)	C425—C424—C427—F472	159.37 (13)

C7—C8—C8A—N1	−177.60 (14)	C423—C424—C427—F472	−22.27 (19)
C7—C8—C8A—C4A	2.1 (2)	C425—C424—C427—F473	38.67 (19)
C5—C4A—C8A—N1	177.86 (13)	C423—C424—C427—F473	−142.98 (13)
C4—C4A—C8A—N1	−0.8 (2)	C425—C424—C427—F471	−80.11 (17)
C5—C4A—C8A—C8	−1.8 (2)	C423—C424—C427—F471	98.24 (16)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C41—H41···O31^{i}	0.95	2.42	3.3290 (17)	161
C426—H426···*Cg*3^{ii}	0.95	3.00	3.7621 (15)	138

Symmetry codes: (i) *x*, *y*+1, *z*; (ii) −*x*+1, *y*−1/2, −*z*+1/2.

Ethyl (*E*)-4-[2-(4-methoxyphenyl)ethenyl]-2-methylquinoline-3-carboxylate (IV)

Crystal data

$C_{22}H_{21}NO_3$
M_r = 347.40
Triclinic, $P\overline{1}$
a = 9.5301 (8) Å
b = 10.3513 (8) Å
c = 10.3621 (8) Å
α = 65.374 (3)°
β = 86.583 (3)°
γ = 76.376 (3)°
V = 902.23 (13) Å^3

Z = 2
$F(000)$ = 368
D_x = 1.279 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 4158 reflections
θ = 2.2–27.5°
μ = 0.09 mm^{-1}
T = 100 K
Needle, yellow
0.30 × 0.12 × 0.05 mm

Data collection

Bruker D8 Venture diffractometer
Radiation source: INCOATEC high brilliance microfocus sealed tube
Multilayer mirror monochromator
φ and ω scans
Absorption correction: multi-scan (SADABS; Bruker, 2016)
T_{min} = 0.954, T_{max} = 0.996

44489 measured reflections
4158 independent reflections
3440 reflections with $I > 2\sigma(I)$
R_{int} = 0.053
θ_{max} = 27.5°, θ_{min} = 2.2°
h = −12→12
k = −13→13
l = −13→13

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.052
$wR(F^2)$ = 0.128
S = 1.07
4158 reflections
238 parameters
0 restraints

Primary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0422P)^2 + 0.7299P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.36 e Å^{-3}
$\Delta\rho_{min}$ = −0.21 e Å^{-3}

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
N1	0.81102 (16)	0.42663 (16)	0.95018 (15)	0.0239 (3)
C2	0.81545 (18)	0.31117 (19)	0.92506 (17)	0.0228 (3)
C3	0.70247 (18)	0.30251 (18)	0.84572 (17)	0.0206 (3)
C4	0.58290 (17)	0.41582 (18)	0.79378 (16)	0.0199 (3)
C4A	0.57445 (18)	0.53964 (18)	0.82549 (16)	0.0204 (3)
C5	0.45312 (19)	0.65895 (18)	0.78548 (17)	0.0242 (4)
H5	0.3727	0.6588	0.7356	0.029*
C6	0.4503 (2)	0.77485 (19)	0.81796 (19)	0.0291 (4)
H6	0.3683	0.8548	0.7901	0.035*
C7	0.5683 (2)	0.7762 (2)	0.89237 (18)	0.0294 (4)
H7	0.5659	0.8576	0.9136	0.035*
C8	0.6858 (2)	0.6619 (2)	0.93411 (18)	0.0272 (4)
H8	0.7647	0.6639	0.9846	0.033*
C8A	0.69172 (18)	0.53980 (19)	0.90306 (16)	0.0221 (3)
C21	0.94750 (19)	0.1883 (2)	0.9796 (2)	0.0312 (4)
H21A	0.9206	0.1020	1.0531	0.047*
H21B	1.0182	0.2176	1.0202	0.047*
H21C	0.9899	0.1649	0.9012	0.047*
C31	0.72030 (17)	0.16500 (19)	0.82311 (18)	0.0221 (3)
O31	0.70662 (14)	0.05033 (13)	0.91571 (13)	0.0296 (3)
O32	0.75597 (14)	0.18596 (14)	0.69062 (13)	0.0286 (3)
C32	0.7718 (2)	0.0610 (2)	0.6539 (2)	0.0325 (4)
H32A	0.8004	−0.0312	0.7402	0.039*
H32B	0.8486	0.0633	0.5848	0.039*
C33	0.6316 (2)	0.0661 (2)	0.59060 (19)	0.0317 (4)
H33A	0.6002	0.1602	0.5089	0.048*
H33B	0.5579	0.0548	0.6621	0.048*
H33C	0.6453	−0.0132	0.5595	0.048*
C41	0.46640 (17)	0.41644 (18)	0.70556 (17)	0.0204 (3)
H41	0.4353	0.5012	0.6202	0.024*
C42	0.40176 (17)	0.30632 (18)	0.73726 (17)	0.0203 (3)
H42	0.4315	0.2241	0.8249	0.024*
C421	0.28969 (17)	0.29870 (18)	0.65120 (16)	0.0193 (3)
C422	0.23677 (18)	0.40931 (19)	0.51900 (17)	0.0230 (3)
H422	0.2688	0.4976	0.4846	0.028*
C423	0.13813 (18)	0.3931 (2)	0.43659 (18)	0.0251 (4)
H423	0.1030	0.4698	0.3469	0.030*
C424	0.09112 (18)	0.2639 (2)	0.48622 (18)	0.0242 (4)
C425	0.13894 (19)	0.1541 (2)	0.61989 (19)	0.0269 (4)

H425	0.1046	0.0671	0.6552	0.032*
C426	0.23657 (18)	0.17209 (18)	0.70116 (18)	0.0232 (3)
H426	0.2681	0.0970	0.7926	0.028*
O424	−0.00165 (14)	0.23441 (16)	0.41192 (13)	0.0334 (3)
C427	−0.0515 (2)	0.3436 (2)	0.2731 (2)	0.0355 (5)
H47A	0.0316	0.3680	0.2155	0.053*
H47B	−0.1105	0.4313	0.2807	0.053*
H47C	−0.1098	0.3061	0.2280	0.053*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.0264 (7)	0.0304 (8)	0.0188 (7)	−0.0121 (6)	−0.0007 (5)	−0.0107 (6)
C2	0.0238 (8)	0.0285 (9)	0.0175 (8)	−0.0093 (7)	−0.0003 (6)	−0.0090 (7)
C3	0.0237 (8)	0.0236 (8)	0.0176 (7)	−0.0087 (6)	0.0008 (6)	−0.0097 (6)
C4	0.0241 (8)	0.0232 (8)	0.0143 (7)	−0.0107 (6)	0.0011 (6)	−0.0070 (6)
C4A	0.0260 (8)	0.0222 (8)	0.0147 (7)	−0.0110 (6)	0.0035 (6)	−0.0068 (6)
C5	0.0306 (9)	0.0222 (8)	0.0178 (8)	−0.0088 (7)	0.0010 (6)	−0.0050 (6)
C6	0.0412 (10)	0.0209 (9)	0.0224 (8)	−0.0072 (7)	0.0024 (7)	−0.0064 (7)
C7	0.0494 (11)	0.0226 (9)	0.0206 (8)	−0.0154 (8)	0.0057 (7)	−0.0100 (7)
C8	0.0395 (10)	0.0307 (9)	0.0177 (8)	−0.0193 (8)	0.0031 (7)	−0.0106 (7)
C8A	0.0289 (9)	0.0263 (8)	0.0142 (7)	−0.0132 (7)	0.0036 (6)	−0.0082 (6)
C21	0.0248 (9)	0.0369 (10)	0.0346 (10)	−0.0043 (8)	−0.0078 (7)	−0.0179 (8)
C31	0.0176 (7)	0.0263 (9)	0.0246 (8)	−0.0047 (6)	−0.0033 (6)	−0.0125 (7)
O31	0.0380 (7)	0.0229 (6)	0.0270 (7)	−0.0067 (5)	−0.0055 (5)	−0.0087 (5)
O32	0.0323 (7)	0.0346 (7)	0.0294 (7)	−0.0138 (6)	0.0065 (5)	−0.0208 (6)
C32	0.0321 (10)	0.0394 (11)	0.0409 (11)	−0.0128 (8)	0.0086 (8)	−0.0297 (9)
C33	0.0440 (11)	0.0314 (10)	0.0246 (9)	−0.0154 (8)	−0.0012 (8)	−0.0126 (8)
C41	0.0221 (8)	0.0227 (8)	0.0161 (7)	−0.0040 (6)	−0.0029 (6)	−0.0079 (6)
C42	0.0205 (8)	0.0231 (8)	0.0168 (7)	−0.0031 (6)	−0.0021 (6)	−0.0086 (6)
C421	0.0187 (7)	0.0231 (8)	0.0180 (7)	−0.0043 (6)	−0.0005 (6)	−0.0104 (6)
C422	0.0250 (8)	0.0256 (9)	0.0188 (8)	−0.0090 (7)	−0.0002 (6)	−0.0078 (7)
C423	0.0235 (8)	0.0317 (9)	0.0180 (8)	−0.0059 (7)	−0.0025 (6)	−0.0082 (7)
C424	0.0186 (8)	0.0366 (10)	0.0221 (8)	−0.0085 (7)	−0.0007 (6)	−0.0155 (7)
C425	0.0264 (9)	0.0286 (9)	0.0276 (9)	−0.0117 (7)	−0.0020 (7)	−0.0103 (7)
C426	0.0235 (8)	0.0234 (8)	0.0204 (8)	−0.0055 (7)	−0.0033 (6)	−0.0064 (7)
O424	0.0319 (7)	0.0486 (8)	0.0252 (7)	−0.0185 (6)	−0.0057 (5)	−0.0150 (6)
C427	0.0259 (9)	0.0599 (13)	0.0257 (9)	−0.0122 (9)	−0.0055 (7)	−0.0205 (9)

Geometric parameters (Å, º)

N1—C2	1.316 (2)	C32—H32A	0.9900
N1—C8A	1.366 (2)	C32—H32B	0.9900
C2—C3	1.434 (2)	C33—H33A	0.9800
C2—C21	1.501 (2)	C33—H33B	0.9800
C3—C4	1.372 (2)	C33—H33C	0.9800
C3—C31	1.506 (2)	C41—C42	1.335 (2)
C4—C4A	1.434 (2)	C41—H41	0.9500

C4—C41	1.477 (2)	C42—C421	1.468 (2)
C4A—C5	1.411 (2)	C42—H42	0.9500
C4A—C8A	1.416 (2)	C421—C422	1.395 (2)
C5—C6	1.369 (2)	C421—C426	1.399 (2)
C5—H5	0.9500	C422—C423	1.389 (2)
C6—C7	1.407 (3)	C422—H422	0.9500
C6—H6	0.9500	C423—C424	1.391 (2)
C7—C8	1.359 (3)	C423—H423	0.9500
C7—H7	0.9500	C424—O424	1.3682 (19)
C8—C8A	1.417 (2)	C424—C425	1.392 (2)
C8—H8	0.9500	C425—C426	1.384 (2)
C21—H21A	0.9800	C425—H425	0.9500
C21—H21B	0.9800	C426—H426	0.9500
C21—H21C	0.9800	O424—C427	1.430 (2)
C31—O31	1.206 (2)	C427—H47A	0.9800
C31—O32	1.334 (2)	C427—H47B	0.9800
O32—C32	1.467 (2)	C427—H47C	0.9800
C32—C33	1.503 (3)		

C2—N1—C8A	118.47 (14)	O32—C32—H32B	109.6
N1—C2—C3	122.42 (16)	C33—C32—H32B	109.6
N1—C2—C21	117.03 (15)	H32A—C32—H32B	108.2
C3—C2—C21	120.52 (15)	C32—C33—H33A	109.5
C4—C3—C2	120.55 (15)	C32—C33—H33B	109.5
C4—C3—C31	121.94 (14)	H33A—C33—H33B	109.5
C2—C3—C31	117.51 (15)	C32—C33—H33C	109.5
C3—C4—C4A	117.41 (14)	H33A—C33—H33C	109.5
C3—C4—C41	123.09 (14)	H33B—C33—H33C	109.5
C4A—C4—C41	119.49 (15)	C42—C41—C4	124.64 (15)
C5—C4A—C8A	118.99 (15)	C42—C41—H41	117.7
C5—C4A—C4	122.73 (15)	C4—C41—H41	117.7
C8A—C4A—C4	118.26 (15)	C41—C42—C421	126.96 (15)
C6—C5—C4A	120.54 (16)	C41—C42—H42	116.5
C6—C5—H5	119.7	C421—C42—H42	116.5
C4A—C5—H5	119.7	C422—C421—C426	117.94 (14)
C5—C6—C7	120.35 (17)	C422—C421—C42	123.34 (15)
C5—C6—H6	119.8	C426—C421—C42	118.68 (14)
C7—C6—H6	119.8	C423—C422—C421	121.39 (16)
C8—C7—C6	120.45 (16)	C423—C422—H422	119.3
C8—C7—H7	119.8	C421—C422—H422	119.3
C6—C7—H7	119.8	C422—C423—C424	119.57 (16)
C7—C8—C8A	120.61 (16)	C422—C423—H423	120.2
C7—C8—H8	119.7	C424—C423—H423	120.2
C8A—C8—H8	119.7	O424—C424—C423	124.44 (16)
N1—C8A—C4A	122.83 (15)	O424—C424—C425	115.64 (16)
N1—C8A—C8	118.15 (15)	C423—C424—C425	119.92 (15)
C4A—C8A—C8	119.02 (16)	C426—C425—C424	119.84 (16)
C2—C21—H21A	109.5	C426—C425—H425	120.1

C2—C21—H21B	109.5	C424—C425—H425	120.1
H21A—C21—H21B	109.5	C425—C426—C421	121.25 (16)
C2—C21—H21C	109.5	C425—C426—H426	119.4
H21A—C21—H21C	109.5	C421—C426—H426	119.4
H21B—C21—H21C	109.5	C424—O424—C427	117.37 (15)
O31—C31—O32	125.09 (15)	O424—C427—H47A	109.5
O31—C31—C3	123.28 (15)	O424—C427—H47B	109.5
O32—C31—C3	111.61 (14)	H47A—C427—H47B	109.5
C31—O32—C32	116.83 (14)	O424—C427—H47C	109.5
O32—C32—C33	110.10 (15)	H47A—C427—H47C	109.5
O32—C32—H32A	109.6	H47B—C427—H47C	109.5
C33—C32—H32A	109.6		

C8A—N1—C2—C3	2.4 (2)	C7—C8—C8A—C4A	1.2 (2)
C8A—N1—C2—C21	−179.40 (15)	C4—C3—C31—O31	−106.0 (2)
N1—C2—C3—C4	−0.8 (3)	C2—C3—C31—O31	73.7 (2)
C21—C2—C3—C4	−178.89 (16)	C4—C3—C31—O32	75.5 (2)
N1—C2—C3—C31	179.59 (15)	C2—C3—C31—O32	−104.85 (17)
C21—C2—C3—C31	1.5 (2)	O31—C31—O32—C32	3.5 (2)
C2—C3—C4—C4A	−1.7 (2)	C3—C31—O32—C32	−178.03 (14)
C31—C3—C4—C4A	177.92 (14)	C31—O32—C32—C33	93.04 (19)
C2—C3—C4—C41	176.96 (15)	C3—C4—C41—C42	48.8 (2)
C31—C3—C4—C41	−3.4 (2)	C4A—C4—C41—C42	−132.53 (17)
C3—C4—C4A—C5	−176.06 (15)	C4—C41—C42—C421	−177.48 (15)
C41—C4—C4A—C5	5.2 (2)	C41—C42—C421—C422	1.2 (3)
C3—C4—C4A—C8A	2.4 (2)	C41—C42—C421—C426	178.77 (16)
C41—C4—C4A—C8A	−176.28 (14)	C426—C421—C422—C423	−2.2 (2)
C8A—C4A—C5—C6	1.7 (2)	C42—C421—C422—C423	175.34 (15)
C4—C4A—C5—C6	−179.86 (16)	C421—C422—C423—C424	−0.2 (3)
C4A—C5—C6—C7	−0.3 (3)	C422—C423—C424—O424	−177.63 (16)
C5—C6—C7—C8	−0.7 (3)	C422—C423—C424—C425	2.3 (3)
C6—C7—C8—C8A	0.2 (3)	O424—C424—C425—C426	177.99 (16)
C2—N1—C8A—C4A	−1.6 (2)	C423—C424—C425—C426	−2.0 (3)
C2—N1—C8A—C8	178.18 (15)	C424—C425—C426—C421	−0.5 (3)
C5—C4A—C8A—N1	177.69 (15)	C422—C421—C426—C425	2.6 (2)
C4—C4A—C8A—N1	−0.9 (2)	C42—C421—C426—C425	−175.09 (15)
C5—C4A—C8A—C8	−2.1 (2)	C423—C424—O424—C427	0.9 (3)
C4—C4A—C8A—C8	179.38 (15)	C425—C424—O424—C427	−179.02 (16)
C7—C8—C8A—N1	−178.61 (16)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C32—H32*B*···O424^{i}	0.99	2.57	3.406 (2)	142
C423—H423···*Cg*1^{ii}	0.95	2.91	3.4894 (19)	120

Symmetry codes: (i) *x*+1, *y*, *z*; (ii) −*x*+1, −*y*+1, −*z*+1.

Ethyl (*E*)-4-[2-(4-bromophenyl)ethenyl]-2-methylquinoline-3-carboxylate (V)

Crystal data

$C_{21}H_{18}BrNO_2$
$M_r = 396.26$
Monoclinic, $P2_1/n$
$a = 9.5709$ (6) Å
$b = 10.6119$ (7) Å
$c = 18.2074$ (10) Å
$\beta = 90.939$ (2)°
$V = 1849.0$ (2) Å^3
$Z = 4$
$F(000) = 808$
$D_x = 1.424$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 4558 reflections
$\theta = 2.2$–$28.3°$
$\mu = 2.24$ mm^{-1}
$T = 100$ K
Block, yellow
0.25 × 0.18 × 0.15 mm

Data collection

Bruker D8 Venture diffractometer
Radiation source: INCOATEC high brilliance microfocus sealed tube
Multilayer mirror monochromator
φ and ω scans
Absorption correction: multi-scan (SADABS; Bruker, 2016)
$T_{min} = 0.595$, $T_{max} = 0.715$
54534 measured reflections
4588 independent reflections
4098 reflections with $I > 2\sigma(I)$
$R_{int} = 0.041$
$\theta_{max} = 28.3°$, $\theta_{min} = 2.2°$
$h = -12 \rightarrow 12$
$k = -14 \rightarrow 14$
$l = -24 \rightarrow 24$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.022$
$wR(F^2) = 0.056$
$S = 1.02$
4588 reflections
228 parameters
0 restraints
Primary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0237P)^2 + 1.0895P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.003$
$\Delta\rho_{max} = 0.35$ e Å^{-3}
$\Delta\rho_{min} = -0.42$ e Å^{-3}

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
N1	0.22794 (12)	0.20653 (11)	0.63602 (6)	0.0186 (2)
C2	0.36010 (14)	0.19499 (13)	0.61835 (7)	0.0179 (3)
C3	0.45699 (14)	0.29687 (13)	0.62494 (7)	0.0161 (2)
C4	0.41542 (14)	0.41110 (12)	0.65325 (7)	0.0156 (2)
C4A	0.27233 (14)	0.42464 (13)	0.67343 (7)	0.0165 (3)
C5	0.21593 (15)	0.53906 (14)	0.70002 (8)	0.0211 (3)
H5	0.2756	0.6092	0.7086	0.025*
C6	0.07589 (16)	0.54916 (15)	0.71338 (8)	0.0249 (3)

H6	0.0390	0.6264	0.7309	0.030*
C7	−0.01353 (16)	0.44582 (15)	0.70130 (9)	0.0261 (3)
H7	−0.1106	0.4540	0.7104	0.031*
C8	0.03799 (15)	0.33360 (14)	0.67658 (8)	0.0225 (3)
H8	−0.0232	0.2641	0.6692	0.027*
C8A	0.18202 (14)	0.32044 (13)	0.66196 (7)	0.0172 (3)
C21	0.40674 (16)	0.06875 (14)	0.59035 (9)	0.0269 (3)
H21A	0.3382	0.0044	0.6036	0.040*
H21B	0.4978	0.0475	0.6124	0.040*
H21C	0.4146	0.0721	0.5368	0.040*
C31	0.60558 (14)	0.27447 (13)	0.60265 (7)	0.0181 (3)
O31	0.70170 (11)	0.25715 (11)	0.64455 (6)	0.0248 (2)
O32	0.61517 (11)	0.27338 (11)	0.52926 (5)	0.0241 (2)
C32	0.75437 (17)	0.25167 (17)	0.49963 (9)	0.0306 (3)
H32A	0.7455	0.2142	0.4500	0.037*
H32B	0.8060	0.1914	0.5315	0.037*
C33	0.83439 (18)	0.37338 (18)	0.49542 (9)	0.0339 (4)
H33A	0.7802	0.4348	0.4666	0.051*
H33B	0.9241	0.3584	0.4718	0.051*
H33C	0.8510	0.4062	0.5451	0.051*
C41	0.51187 (14)	0.51853 (12)	0.66208 (7)	0.0168 (3)
H41	0.5177	0.5591	0.7086	0.020*
C42	0.59134 (15)	0.56169 (13)	0.60807 (7)	0.0185 (3)
H42	0.5825	0.5209	0.5618	0.022*
C421	0.69122 (14)	0.66634 (13)	0.61359 (7)	0.0174 (3)
C422	0.73110 (15)	0.72094 (13)	0.68058 (7)	0.0189 (3)
H422	0.6918	0.6908	0.7248	0.023*
C423	0.82710 (14)	0.81836 (13)	0.68316 (7)	0.0188 (3)
H423	0.8536	0.8552	0.7289	0.023*
C424	0.88435 (14)	0.86183 (12)	0.61828 (7)	0.0176 (3)
Br42	1.02007 (2)	0.99228 (2)	0.62329 (2)	0.02109 (5)
C425	0.84705 (16)	0.81022 (14)	0.55109 (8)	0.0235 (3)
H425	0.8861	0.8412	0.5070	0.028*
C426	0.75115 (16)	0.71199 (14)	0.54958 (8)	0.0235 (3)
H426	0.7257	0.6750	0.5038	0.028*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.0177 (6)	0.0175 (5)	0.0206 (6)	−0.0064 (4)	0.0008 (4)	0.0001 (4)
C2	0.0191 (7)	0.0169 (6)	0.0178 (6)	−0.0046 (5)	0.0003 (5)	−0.0006 (5)
C3	0.0147 (6)	0.0187 (6)	0.0149 (6)	−0.0045 (5)	0.0009 (5)	0.0007 (5)
C4	0.0158 (6)	0.0170 (6)	0.0138 (6)	−0.0049 (5)	−0.0018 (5)	0.0018 (5)
C4A	0.0160 (6)	0.0180 (6)	0.0156 (6)	−0.0034 (5)	−0.0003 (5)	0.0008 (5)
C5	0.0200 (7)	0.0199 (6)	0.0233 (7)	−0.0036 (5)	−0.0003 (5)	−0.0028 (5)
C6	0.0212 (7)	0.0262 (7)	0.0274 (7)	0.0023 (6)	−0.0001 (6)	−0.0064 (6)
C7	0.0161 (7)	0.0326 (8)	0.0295 (8)	−0.0016 (6)	0.0020 (6)	−0.0028 (6)
C8	0.0159 (7)	0.0265 (7)	0.0251 (7)	−0.0070 (5)	0.0008 (5)	−0.0011 (6)

C8A	0.0155 (6)	0.0195 (6)	0.0167 (6)	−0.0045 (5)	0.0007 (5)	0.0012 (5)
C21	0.0246 (8)	0.0188 (7)	0.0374 (8)	−0.0057 (6)	0.0054 (6)	−0.0077 (6)
C31	0.0179 (6)	0.0169 (6)	0.0194 (6)	−0.0048 (5)	0.0019 (5)	−0.0020 (5)
O31	0.0167 (5)	0.0347 (6)	0.0231 (5)	−0.0001 (4)	−0.0004 (4)	0.0009 (4)
O32	0.0202 (5)	0.0344 (6)	0.0178 (5)	−0.0032 (4)	0.0034 (4)	−0.0043 (4)
C32	0.0250 (8)	0.0413 (9)	0.0258 (8)	−0.0002 (7)	0.0109 (6)	−0.0066 (7)
C33	0.0262 (8)	0.0466 (10)	0.0293 (8)	−0.0023 (7)	0.0084 (7)	0.0050 (7)
C41	0.0153 (6)	0.0167 (6)	0.0184 (6)	−0.0038 (5)	−0.0019 (5)	−0.0004 (5)
C42	0.0202 (7)	0.0185 (6)	0.0165 (6)	−0.0070 (5)	−0.0027 (5)	−0.0003 (5)
C421	0.0171 (6)	0.0174 (6)	0.0175 (6)	−0.0054 (5)	−0.0013 (5)	0.0010 (5)
C422	0.0201 (7)	0.0206 (6)	0.0162 (6)	−0.0066 (5)	0.0028 (5)	−0.0002 (5)
C423	0.0191 (7)	0.0194 (6)	0.0179 (6)	−0.0068 (5)	0.0011 (5)	−0.0029 (5)
C424	0.0160 (6)	0.0158 (6)	0.0211 (6)	−0.0069 (5)	0.0003 (5)	0.0003 (5)
Br42	0.02136 (8)	0.02079 (8)	0.02118 (7)	−0.01179 (5)	0.00231 (5)	−0.00103 (5)
C425	0.0268 (8)	0.0264 (7)	0.0174 (6)	−0.0125 (6)	0.0020 (6)	0.0032 (5)
C426	0.0286 (8)	0.0261 (7)	0.0158 (6)	−0.0132 (6)	−0.0015 (5)	−0.0004 (5)

Geometric parameters (Å, º)

N1—C2	1.3160 (18)	O32—C32	1.4638 (17)
N1—C8A	1.3725 (18)	C32—C33	1.504 (2)
C2—C3	1.4282 (18)	C32—H32A	0.9900
C2—C21	1.5038 (19)	C32—H32B	0.9900
C3—C4	1.3784 (19)	C33—H33A	0.9800
C3—C31	1.5042 (19)	C33—H33B	0.9800
C4—C4A	1.4309 (18)	C33—H33C	0.9800
C4—C41	1.4741 (18)	C41—C42	1.3341 (19)
C4A—C8A	1.4168 (18)	C41—H41	0.9500
C4A—C5	1.4173 (19)	C42—C421	1.4676 (18)
C5—C6	1.370 (2)	C42—H42	0.9500
C5—H5	0.9500	C421—C426	1.3942 (19)
C6—C7	1.406 (2)	C421—C422	1.3977 (18)
C6—H6	0.9500	C422—C423	1.3834 (18)
C7—C8	1.368 (2)	C422—H422	0.9500
C7—H7	0.9500	C423—C424	1.3891 (18)
C8—C8A	1.4152 (19)	C423—H423	0.9500
C8—H8	0.9500	C424—C425	1.3820 (19)
C21—H21A	0.9800	C424—Br42	1.8996 (13)
C21—H21B	0.9800	C425—C426	1.3889 (19)
C21—H21C	0.9800	C425—H425	0.9500
C31—O31	1.1997 (17)	C426—H426	0.9500
C31—O32	1.3409 (17)		

C2—N1—C8A	118.72 (12)	C31—O32—C32	116.51 (12)
N1—C2—C3	122.30 (13)	O32—C32—C33	110.54 (13)
N1—C2—C21	117.24 (12)	O32—C32—H32A	109.5
C3—C2—C21	120.46 (12)	C33—C32—H32A	109.5
C4—C3—C2	120.41 (12)	O32—C32—H32B	109.5

C4—C3—C31	121.32 (12)	C33—C32—H32B	109.5
C2—C3—C31	118.24 (12)	H32A—C32—H32B	108.1
C3—C4—C4A	117.88 (12)	C32—C33—H33A	109.5
C3—C4—C41	122.47 (12)	C32—C33—H33B	109.5
C4A—C4—C41	119.64 (12)	H33A—C33—H33B	109.5
C8A—C4A—C5	118.96 (12)	C32—C33—H33C	109.5
C8A—C4A—C4	117.89 (12)	H33A—C33—H33C	109.5
C5—C4A—C4	123.04 (12)	H33B—C33—H33C	109.5
C6—C5—C4A	120.45 (13)	C42—C41—C4	123.28 (12)
C6—C5—H5	119.8	C42—C41—H41	118.4
C4A—C5—H5	119.8	C4—C41—H41	118.4
C5—C6—C7	120.38 (14)	C41—C42—C421	126.00 (13)
C5—C6—H6	119.8	C41—C42—H42	117.0
C7—C6—H6	119.8	C421—C42—H42	117.0
C8—C7—C6	120.58 (14)	C426—C421—C422	118.37 (12)
C8—C7—H7	119.7	C426—C421—C42	118.82 (12)
C6—C7—H7	119.7	C422—C421—C42	122.80 (12)
C7—C8—C8A	120.35 (13)	C423—C422—C421	120.74 (12)
C7—C8—H8	119.8	C423—C422—H422	119.6
C8A—C8—H8	119.8	C421—C422—H422	119.6
N1—C8A—C8	118.03 (12)	C422—C423—C424	119.36 (12)
N1—C8A—C4A	122.69 (12)	C422—C423—H423	120.3
C8—C8A—C4A	119.27 (13)	C424—C423—H423	120.3
C2—C21—H21A	109.5	C425—C424—C423	121.43 (12)
C2—C21—H21B	109.5	C425—C424—Br42	119.86 (10)
H21A—C21—H21B	109.5	C423—C424—Br42	118.69 (10)
C2—C21—H21C	109.5	C424—C425—C426	118.42 (13)
H21A—C21—H21C	109.5	C424—C425—H425	120.8
H21B—C21—H21C	109.5	C426—C425—H425	120.8
O31—C31—O32	124.62 (13)	C425—C426—C421	121.67 (13)
O31—C31—C3	124.83 (12)	C425—C426—H426	119.2
O32—C31—C3	110.53 (12)	C421—C426—H426	119.2

C8A—N1—C2—C3	−0.3 (2)	C5—C4A—C8A—C8	0.6 (2)
C8A—N1—C2—C21	179.82 (13)	C4—C4A—C8A—C8	−175.68 (12)
N1—C2—C3—C4	2.6 (2)	C4—C3—C31—O31	75.39 (19)
C21—C2—C3—C4	−177.51 (13)	C2—C3—C31—O31	−102.56 (17)
N1—C2—C3—C31	−179.45 (12)	C4—C3—C31—O32	−106.17 (14)
C21—C2—C3—C31	0.46 (19)	C2—C3—C31—O32	75.88 (15)
C2—C3—C4—C4A	−1.82 (19)	O31—C31—O32—C32	−1.2 (2)
C31—C3—C4—C4A	−179.73 (12)	C3—C31—O32—C32	−179.65 (12)
C2—C3—C4—C41	179.17 (12)	C31—O32—C32—C33	−85.21 (17)
C31—C3—C4—C41	1.3 (2)	C3—C4—C41—C42	51.4 (2)
C3—C4—C4A—C8A	−0.97 (18)	C4A—C4—C41—C42	−127.57 (15)
C41—C4—C4A—C8A	178.07 (12)	C4—C41—C42—C421	−178.74 (13)
C3—C4—C4A—C5	−177.12 (13)	C41—C42—C421—C426	−170.67 (15)
C41—C4—C4A—C5	1.9 (2)	C41—C42—C421—C422	10.6 (2)
C8A—C4A—C5—C6	−0.9 (2)	C426—C421—C422—C423	0.4 (2)

C4—C4A—C5—C6	175.18 (13)	C42—C421—C422—C423	179.12 (13)
C4A—C5—C6—C7	0.4 (2)	C421—C422—C423—C424	−0.1 (2)
C5—C6—C7—C8	0.5 (2)	C422—C423—C424—C425	0.3 (2)
C6—C7—C8—C8A	−0.8 (2)	C422—C423—C424—Br42	−178.17 (11)
C2—N1—C8A—C8	176.31 (13)	C423—C424—C425—C426	−0.7 (2)
C2—N1—C8A—C4A	−2.7 (2)	Br42—C424—C425—C426	177.77 (12)
C7—C8—C8A—N1	−178.88 (14)	C424—C425—C426—C421	0.9 (2)
C7—C8—C8A—C4A	0.2 (2)	C422—C421—C426—C425	−0.8 (2)
C5—C4A—C8A—N1	179.67 (13)	C42—C421—C426—C425	−179.57 (14)
C4—C4A—C8A—N1	3.4 (2)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C423—H423···O31^{i}	0.95	2.59	3.2197 (17)	124
C426—H426···*Cg*1^{ii}	0.95	2.74	3.5961 (16)	151

Symmetry codes: (i) −*x*+3/2, *y*+1/2, −*z*+3/2; (ii) −*x*+1, −*y*+1, −*z*+1.

Ethyl (*E*)-2-methyl-4-{2-[4-(trifluoromethyl)phenyl]ethenyl}quinoline-3-carboxylate (VI)

Crystal data

$C_{22}H_{18}F_3NO_2$
M_r = 385.37
Triclinic, $P\overline{1}$
a = 8.7465 (10) Å
b = 9.9436 (11) Å
c = 11.1116 (11) Å
α = 105.446 (4)°
β = 99.763 (4)°
γ = 97.204 (4)°
V = 903.08 (17) Å^3
Z = 2
$F(000)$ = 400
D_x = 1.417 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 4022 reflections
θ = 2.2–28.3°
μ = 0.11 mm^{-1}
T = 100 K
Block, yellow
0.27 × 0.20 × 0.18 mm

Data collection

Bruker D8 Venture diffractometer
Radiation source: INCOATEC high brilliance microfocus sealed tube
Multilayer mirror monochromator
φ and ω scans
Absorption correction: multi-scan (SADABS; Bruker, 2016)
T_{min} = 0.954, T_{max} = 0.980
59876 measured reflections
4491 independent reflections
3692 reflections with $I > 2\sigma(I)$
R_{int} = 0.042
θ_{max} = 28.3°, θ_{min} = 2.2°
h = −11→11
k = −13→13
l = −14→14

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.044
$wR(F^2)$ = 0.120
S = 1.05
4491 reflections
255 parameters
0 restraints
Primary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0452P)^2 + 0.7003P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$

$\Delta\rho_{max}$ = 0.43 e Å^{-3} $\Delta\rho_{min}$ = −0.43 e Å^{-3}

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
N1	0.07789 (14)	0.22664 (13)	0.15945 (11)	0.0204 (2)
C2	0.07976 (16)	0.36156 (15)	0.21549 (13)	0.0192 (3)
C3	0.16984 (15)	0.43512 (14)	0.34110 (13)	0.0171 (3)
C4	0.25843 (15)	0.36523 (14)	0.41074 (12)	0.0168 (3)
C4A	0.26008 (15)	0.21910 (14)	0.35031 (13)	0.0173 (3)
C5	0.34837 (17)	0.13520 (15)	0.40993 (14)	0.0216 (3)
H5	0.4093	0.1758	0.4944	0.026*
C6	0.34646 (18)	−0.00371 (16)	0.34678 (15)	0.0251 (3)
H6	0.4067	−0.0585	0.3877	0.030*
C7	0.25608 (19)	−0.06624 (15)	0.22177 (14)	0.0250 (3)
H7	0.2562	−0.1626	0.1789	0.030*
C8	0.16816 (18)	0.01086 (15)	0.16161 (14)	0.0232 (3)
H8	0.1067	−0.0323	0.0776	0.028*
C8A	0.16872 (16)	0.15504 (14)	0.22455 (13)	0.0187 (3)
C21	−0.01256 (19)	0.43927 (16)	0.13843 (14)	0.0251 (3)
H21A	−0.0605	0.3749	0.0530	0.038*
H21B	0.0581	0.5199	0.1310	0.038*
H21C	−0.0955	0.4737	0.1810	0.038*
C31	0.17896 (16)	0.59297 (14)	0.38398 (13)	0.0180 (3)
O31	0.29053 (12)	0.67679 (11)	0.38197 (10)	0.0231 (2)
O32	0.04628 (11)	0.62833 (10)	0.41672 (9)	0.0200 (2)
C32	0.03379 (17)	0.77805 (14)	0.44196 (14)	0.0207 (3)
H32A	−0.0788	0.7871	0.4320	0.025*
H32B	0.0773	0.8161	0.3786	0.025*
C33	0.12155 (18)	0.86413 (16)	0.57495 (14)	0.0250 (3)
H33A	0.0848	0.8219	0.6375	0.037*
H33B	0.1019	0.9616	0.5917	0.037*
H33C	0.2348	0.8646	0.5818	0.037*
C41	0.35128 (16)	0.43372 (14)	0.54207 (13)	0.0184 (3)
H41	0.4482	0.4038	0.5650	0.022*
C42	0.31086 (16)	0.53430 (15)	0.63157 (13)	0.0193 (3)
H42	0.2140	0.5648	0.6098	0.023*
C421	0.40671 (16)	0.60055 (15)	0.76133 (13)	0.0192 (3)
C422	0.52605 (17)	0.53948 (15)	0.81430 (13)	0.0213 (3)
H422	0.5451	0.4509	0.7667	0.026*
C423	0.61718 (17)	0.60661 (16)	0.93570 (14)	0.0236 (3)
H423	0.6985	0.5643	0.9707	0.028*
C424	0.58945 (17)	0.73547 (16)	1.00582 (13)	0.0228 (3)

C425	0.46796 (19)	0.79605 (16)	0.95666 (14)	0.0259 (3)
H425	0.4474	0.8832	1.0059	0.031*
C426	0.37692 (18)	0.72844 (16)	0.83523 (14)	0.0240 (3)
H426	0.2933	0.7695	0.8018	0.029*
C427	0.6902 (2)	0.81210 (18)	1.13510 (15)	0.0302 (3)
F471	0.74662 (16)	0.94629 (12)	1.14494 (11)	0.0523 (3)
F472	0.81434 (15)	0.75355 (14)	1.16419 (11)	0.0539 (4)
F473	0.61164 (15)	0.81641 (14)	1.22927 (10)	0.0506 (3)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.0231 (6)	0.0206 (6)	0.0159 (5)	0.0032 (4)	0.0012 (4)	0.0049 (4)
C2	0.0200 (6)	0.0198 (6)	0.0175 (6)	0.0032 (5)	0.0025 (5)	0.0061 (5)
C3	0.0170 (6)	0.0167 (6)	0.0178 (6)	0.0030 (5)	0.0044 (5)	0.0050 (5)
C4	0.0153 (6)	0.0176 (6)	0.0164 (6)	0.0014 (5)	0.0026 (5)	0.0045 (5)
C4A	0.0178 (6)	0.0166 (6)	0.0177 (6)	0.0027 (5)	0.0037 (5)	0.0054 (5)
C5	0.0227 (7)	0.0206 (7)	0.0203 (6)	0.0039 (5)	0.0015 (5)	0.0061 (5)
C6	0.0291 (7)	0.0198 (7)	0.0263 (7)	0.0070 (6)	0.0018 (6)	0.0079 (6)
C7	0.0311 (8)	0.0170 (6)	0.0250 (7)	0.0051 (6)	0.0050 (6)	0.0036 (5)
C8	0.0286 (7)	0.0201 (7)	0.0181 (6)	0.0030 (5)	0.0032 (5)	0.0023 (5)
C8A	0.0201 (6)	0.0187 (6)	0.0169 (6)	0.0028 (5)	0.0034 (5)	0.0053 (5)
C21	0.0292 (7)	0.0249 (7)	0.0196 (7)	0.0059 (6)	−0.0017 (5)	0.0080 (5)
C31	0.0192 (6)	0.0183 (6)	0.0166 (6)	0.0044 (5)	0.0024 (5)	0.0055 (5)
O31	0.0212 (5)	0.0190 (5)	0.0293 (5)	0.0034 (4)	0.0061 (4)	0.0070 (4)
O32	0.0183 (5)	0.0184 (5)	0.0230 (5)	0.0045 (4)	0.0043 (4)	0.0051 (4)
C32	0.0216 (6)	0.0184 (6)	0.0225 (7)	0.0072 (5)	0.0035 (5)	0.0057 (5)
C33	0.0271 (7)	0.0217 (7)	0.0236 (7)	0.0065 (6)	0.0028 (6)	0.0032 (5)
C41	0.0165 (6)	0.0186 (6)	0.0182 (6)	0.0005 (5)	0.0004 (5)	0.0055 (5)
C42	0.0181 (6)	0.0204 (6)	0.0177 (6)	0.0023 (5)	0.0011 (5)	0.0050 (5)
C421	0.0183 (6)	0.0207 (6)	0.0167 (6)	0.0015 (5)	0.0026 (5)	0.0038 (5)
C422	0.0215 (7)	0.0219 (7)	0.0191 (6)	0.0050 (5)	0.0034 (5)	0.0037 (5)
C423	0.0220 (7)	0.0288 (7)	0.0196 (7)	0.0068 (6)	0.0018 (5)	0.0069 (6)
C424	0.0234 (7)	0.0246 (7)	0.0164 (6)	0.0007 (5)	0.0013 (5)	0.0027 (5)
C425	0.0307 (8)	0.0207 (7)	0.0214 (7)	0.0047 (6)	0.0012 (6)	0.0004 (5)
C426	0.0250 (7)	0.0225 (7)	0.0212 (7)	0.0063 (5)	0.0001 (5)	0.0030 (5)
C427	0.0324 (8)	0.0313 (8)	0.0199 (7)	0.0023 (6)	−0.0023 (6)	0.0023 (6)
F471	0.0664 (8)	0.0348 (6)	0.0344 (6)	−0.0146 (5)	−0.0164 (5)	0.0025 (5)
F472	0.0479 (7)	0.0611 (8)	0.0335 (6)	0.0201 (6)	−0.0186 (5)	−0.0060 (5)
F473	0.0511 (7)	0.0703 (8)	0.0182 (5)	−0.0011 (6)	0.0044 (4)	−0.0003 (5)

Geometric parameters (Å, º)

N1—C2	1.3168 (18)	C32—C33	1.511 (2)
N1—C8A	1.3720 (18)	C32—H32A	0.9900
C2—C3	1.4292 (18)	C32—H32B	0.9900
C2—C21	1.5021 (19)	C33—H33A	0.9800
C3—C4	1.3786 (18)	C33—H33B	0.9800

C3—C31	1.5014 (18)	C33—H33C	0.9800
C4—C4A	1.4322 (18)	C41—C42	1.3388 (19)
C4—C41	1.4774 (18)	C41—H41	0.9500
C4A—C5	1.4191 (19)	C42—C421	1.4701 (18)
C4A—C8A	1.4200 (18)	C42—H42	0.9500
C5—C6	1.369 (2)	C421—C422	1.3949 (19)
C5—H5	0.9500	C421—C426	1.400 (2)
C6—C7	1.409 (2)	C422—C423	1.3867 (19)
C6—H6	0.9500	C422—H422	0.9500
C7—C8	1.368 (2)	C423—C424	1.385 (2)
C7—H7	0.9500	C423—H423	0.9500
C8—C8A	1.4168 (19)	C424—C425	1.389 (2)
C8—H8	0.9500	C424—C427	1.497 (2)
C21—H21A	0.9800	C425—C426	1.387 (2)
C21—H21B	0.9800	C425—H425	0.9500
C21—H21C	0.9800	C426—H426	0.9500
C31—O31	1.2087 (17)	C427—F472	1.328 (2)
C31—O32	1.3342 (17)	C427—F471	1.332 (2)
O32—C32	1.4614 (16)	C427—F473	1.340 (2)

C2—N1—C8A	118.08 (12)	C33—C32—H32A	109.3
N1—C2—C3	122.90 (12)	O32—C32—H32B	109.3
N1—C2—C21	116.74 (12)	C33—C32—H32B	109.3
C3—C2—C21	120.30 (12)	H32A—C32—H32B	108.0
C4—C3—C2	120.52 (12)	C32—C33—H33A	109.5
C4—C3—C31	122.57 (12)	C32—C33—H33B	109.5
C2—C3—C31	116.42 (11)	H33A—C33—H33B	109.5
C3—C4—C4A	117.29 (12)	C32—C33—H33C	109.5
C3—C4—C41	123.40 (12)	H33A—C33—H33C	109.5
C4A—C4—C41	119.30 (12)	H33B—C33—H33C	109.5
C5—C4A—C8A	118.41 (12)	C42—C41—C4	125.72 (13)
C5—C4A—C4	123.25 (12)	C42—C41—H41	117.1
C8A—C4A—C4	118.35 (12)	C4—C41—H41	117.1
C6—C5—C4A	120.57 (13)	C41—C42—C421	124.22 (13)
C6—C5—H5	119.7	C41—C42—H42	117.9
C4A—C5—H5	119.7	C421—C42—H42	117.9
C5—C6—C7	120.64 (13)	C422—C421—C426	118.55 (13)
C5—C6—H6	119.7	C422—C421—C42	122.19 (13)
C7—C6—H6	119.7	C426—C421—C42	119.26 (13)
C8—C7—C6	120.48 (13)	C423—C422—C421	120.76 (13)
C8—C7—H7	119.8	C423—C422—H422	119.6
C6—C7—H7	119.8	C421—C422—H422	119.6
C7—C8—C8A	120.02 (13)	C424—C423—C422	119.87 (13)
C7—C8—H8	120.0	C424—C423—H423	120.1
C8A—C8—H8	120.0	C422—C423—H423	120.1
N1—C8A—C8	117.31 (12)	C423—C424—C425	120.37 (13)
N1—C8A—C4A	122.82 (12)	C423—C424—C427	120.69 (14)
C8—C8A—C4A	119.87 (12)	C425—C424—C427	118.94 (14)

C2—C21—H21A	109.5	C426—C425—C424	119.56 (14)
C2—C21—H21B	109.5	C426—C425—H425	120.2
H21A—C21—H21B	109.5	C424—C425—H425	120.2
C2—C21—H21C	109.5	C425—C426—C421	120.83 (14)
H21A—C21—H21C	109.5	C425—C426—H426	119.6
H21B—C21—H21C	109.5	C421—C426—H426	119.6
O31—C31—O32	124.46 (13)	F472—C427—F471	106.29 (15)
O31—C31—C3	123.44 (12)	F472—C427—F473	105.83 (14)
O32—C31—C3	111.98 (11)	F471—C427—F473	106.37 (14)
C31—O32—C32	116.34 (11)	F472—C427—C424	113.19 (14)
O32—C32—C33	111.65 (11)	F471—C427—C424	111.94 (13)
O32—C32—H32A	109.3	F473—C427—C424	112.70 (14)

C8A—N1—C2—C3	−1.0 (2)	C2—C3—C31—O31	−99.75 (16)
C8A—N1—C2—C21	176.13 (12)	C4—C3—C31—O32	−111.53 (14)
N1—C2—C3—C4	−1.0 (2)	C2—C3—C31—O32	76.46 (15)
C21—C2—C3—C4	−178.09 (13)	O31—C31—O32—C32	4.82 (19)
N1—C2—C3—C31	171.16 (13)	C3—C31—O32—C32	−171.35 (11)
C21—C2—C3—C31	−5.91 (19)	C31—O32—C32—C33	−80.77 (15)
C2—C3—C4—C4A	2.11 (19)	C3—C4—C41—C42	34.8 (2)
C31—C3—C4—C4A	−169.59 (12)	C4A—C4—C41—C42	−145.85 (14)
C2—C3—C4—C41	−178.53 (12)	C4—C41—C42—C421	−179.75 (12)
C31—C3—C4—C41	9.8 (2)	C41—C42—C421—C422	−17.7 (2)
C3—C4—C4A—C5	178.82 (13)	C41—C42—C421—C426	162.95 (14)
C41—C4—C4A—C5	−0.6 (2)	C426—C421—C422—C423	−2.4 (2)
C3—C4—C4A—C8A	−1.24 (19)	C42—C421—C422—C423	178.24 (13)
C41—C4—C4A—C8A	179.38 (12)	C421—C422—C423—C424	0.3 (2)
C8A—C4A—C5—C6	0.7 (2)	C422—C423—C424—C425	1.8 (2)
C4—C4A—C5—C6	−179.36 (13)	C422—C423—C424—C427	−177.82 (14)
C4A—C5—C6—C7	−0.4 (2)	C423—C424—C425—C426	−1.7 (2)
C5—C6—C7—C8	−0.3 (2)	C427—C424—C425—C426	177.92 (14)
C6—C7—C8—C8A	0.6 (2)	C424—C425—C426—C421	−0.5 (2)
C2—N1—C8A—C8	−178.55 (13)	C422—C421—C426—C425	2.5 (2)
C2—N1—C8A—C4A	1.9 (2)	C42—C421—C426—C425	−178.13 (14)
C7—C8—C8A—N1	−179.83 (14)	C423—C424—C427—F472	8.1 (2)
C7—C8—C8A—C4A	−0.3 (2)	C425—C424—C427—F472	−171.53 (15)
C5—C4A—C8A—N1	179.16 (13)	C423—C424—C427—F471	128.18 (17)
C4—C4A—C8A—N1	−0.8 (2)	C425—C424—C427—F471	−51.4 (2)
C5—C4A—C8A—C8	−0.3 (2)	C423—C424—C427—F473	−111.97 (17)
C4—C4A—C8A—C8	179.70 (12)	C425—C424—C427—F473	68.4 (2)
C4—C3—C31—O31	72.26 (19)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C41—H41···O31^{i}	0.95	2.54	3.4922 (18)	178

C422—H422···O31^{i}	0.95	2.56	3.3924 (19)	146

Symmetry code: (i) −*x*+1, −*y*+1, −*z*+1.